ALTERNATIVE METHODS FOR DETERMINING THE BIOCHEMICAL METHANE POTENTIAL OF MUNICIPAL SOLID WASTES

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Abstract

The biochemical methane potential (BMP) of a substance is a measure of the volume of methane gas produced per unit mass of that substance, through the process of anaerobic biodegradation. As the socio-economic consequences of climate change have become more apparent, the ability to predict the long-term cumulative environmental impact of various human activities has become more necessary. Landfills can be a substantial source of methane (a greenhouse gas) to the atmosphere, and consequently BMP is an important tool for predicting the potential cumulative long-term impacts of a landfill to the environment. From a strictly economic perspective, the practice of landfill methane extraction for industrial uses is becoming much more common. In this case, BMP is an important tool for predicting the economic feasibility of such a project.

Current methods for determining the BMP of municipal solid wastes (MSW) are both time-consuming and inconsistent. A review of literature on the topic yields many different descriptions of the test, with large variations in sample sizes, incubation times, procedures, etc. Most of these methods also require expensive, and specialized equipment. This thesis describes a simple approach to the BMP test that might be carried out in a variety of laboratory settings, such as an on-site lab equipped with basic, simple, and inexpensive equipment. The method relies on a much larger than typical sample mass to produce large volumes of gas that are measured for composition multiple times over the course of the test. The volume and composition data is then used to produce a cumulative methane potential curve which can be fitted to a first-order decay model in order to predict an ultimate BMP value. The taking of multiple measurements on large volumes of gas, allows for the use of a portable field instrument called the GEM^{TM}2000 to measure gas composition. By fitting the data to a curve in order to determine ultimate methane potential, individual measurement errors are averaged out and the final result has a precision similar to more traditional BMP methods, which rely on bulky and expensive gas chromatographs.

Testing has been conducted on MSW samples from 3 separate sites. The method used involved comparatively large samples of waste (~200 g) and no limit was set on incubation time. The use of large waste samples produces large quantities of gas that must be collected and analyzed often. The method provided favourable results, consistent within acceptable limits of variability when compared with other BMP methods. There is even some evidence suggesting that the use of large waste samples improves the accuracy of the test, despite the use of equipment which provides less precise measurements of gas concentration.

Given the long duration required for testing, the results were also evaluated for possible correlations between loss on ignition data and specific gravity measurements; two simple tests that can be conducted rapidly. Both data sets show a rough correlation with BMP, and may be used to quickly estimate ultimate BMP values, but the loss on ignition relationship provides the better correlation. Lastly, initial steps were taken in the development of what has been dubbed the Biochemical CO_2 Potential (BCP) test, taking advantage of the relatively quicker rate of aerobic degradation. There are preliminary indications that the BCP method may be a viable alternative to the BMP, but the data set so far is small and further research is required to confirm that hypothesis.

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List of Abbreviations

BCP	Biochemical CO_2 Potential
BMP	Biochemical Methane Potential
BOD	Biochemical Oxygen Demand
CV	Coefficient of Variation
GC	Gas Chromatograph
LOI	Loss On Ignition
MSW	Municipal Solid Waste
NCSU	North Carolina State University
RMWB	Rural Municipality of Wood Buffalo
VS	Volatile Solids

Chapter 1 Introduction

1.1. General

The time for complete degradation of municipal solid waste (MSW) landfills lasts on the order of decades, and can extend well beyond 100 years (Knox et al. 2011). The main products of the anaerobic degradation process that occurs within landfills are carbon dioxide (CO_2) and methane (CH_4), which are produced in roughly equal quantities. Of anthropogenic greenhouse gases, these also happen to be the two that have, respectively, the first and second greatest impacts on climate change (IPCC, 1996). In the field of landfill studies, methane is the gas of most interest because of its usefulness as a fuel for various industrial activities, but also because of its much larger greenhouse gas potential, which is approximately 25 times greater than an equivalent mass of CO_2 over a 100 year period (IPCC, 2007). Because of this, it is often desirable for engineers and/or landfill operators to have some idea of the cumulative volume of methane that will be produced by a mass of waste buried within a landfill. To do this we use a method known as the biochemical methane potential (BMP) test.

1.2. Biochemical Methane Potential

The biochemical methane potential of a substance is a measure of the volume of methane gas produced per unit mass of that substance, through the process of anaerobic biodegradation. As the socio-economic consequences of climate change have become more apparent, the ability to predict the environmental impact of various human activities has become more necessary. The BMP is an important tool for helping to predict those consequences.

Regardless of the environmental issues, there is also a strictly economic incentive for wanting to predict methane volumes within waste. Landfills are a relatively cheap and convenient source of methane gas. In a large enough landfill, the amount of the gas that may be recovered often has a value exceeding the cost of extracting it.

1.3. Need for improved BMP Test

The BMP test is typically performed by anaerobically digesting a samll mass of waste inside a sealed bottle. The sample is typically inoculated with a seed of anaerobic microorganisms to kickstart the digestion process. Sometimes a nutrient media is also added to ensure a healthy environment for microbial activity. After a predetermined amount of time the excess gas produced is removed from the vessel and analyzed for composition. The measured volume and compositon of gas is then divided by the known mass of the sample to produce a methane potential.

As it is currently practiced, BMP testing requires a lengthy period of time. With the most commonly used methods, incubation times per vessel can vary from 21 to 150+ days. For many projects, this prolonged testing period can be a deterrent to performing the test. Another issue is the requirement for sophisticated, and expensive equipment. Almost all BMP testing to date is conducted using a gas chromatograph (GC) to measure gas compositions (Raposo et al. 2011). GCs are bulky and sensitive pieces of equipment that cannot be easily transported, and require trained operators if they are to be used effectively. In addition to these undesirable requirements, the BMP test still has a relatively low repeatability. Given the increasing importance of measuring the long-term cumulative greenhouse gas emissions associated with waste, there is a need to address these issues and attempt to make the process more accessible to less specialized labs and technicians.

Another problem with BMP testing is the lack of any accepted standard method. It seems that every laboratory performing BMP tests develops its own unique method and procedure. An interlaboratory study on BMP methods, published in 2011 by Raposo et al., outlined many differences in methodology that could possibly effect the results. Some of these include:

- inoculum used (source, age, amount, etc.);
- substrate used (source, particle size, amount etc.);
- gas handling (gas[es] measured, method of volume/composition measurements, etc.); and,
- physical conditions (reactor volume, temperature, allotted time, etc.).

These differences are understandable, given the wide range of substrates that BMP testing has been performed on, but makes it difficult to evaluate the performance of different methods. The problem is only exacerbated when testing an extremely variable substrate like MSW.

1.4. Objectives of the Research

The objectives of this research are to provide alternative methods or approaches to the BMP test that can improve it in one or more of the following areas:

1. time requirement;
2. repeatability; and,
3. need for specialized equipment.

1.5. Scope of the Research

It is unlikely that any one alternative method will provide improvement in all three areas listed above. For the purposes of this thesis, any alternative method that provides an improvement in at least one area will be considered an improved test method. The idea is that providing options for researchers and technicians will allow them to choose the method most appropriate for the constraints and requirements of their project.

Three alternate approaches were explored in attempting to improve upon the existing test method. They are as follows:

1. make minor modifications to the procedure and/or apparatus of the existing test method;
2. correlate the results of the existing method to a simpler test or tests; or,
3. develop an entirely novel test method from scratch.

A version of the BMP method using a much larger sample mass is developed and hopes to improve on repeatability while requiring only basic equipment. Also, possible correlations between BMP with loss on ignition (LOI) and specific gravity are investigated. If successful, these correlations may provide methods of estimating BMP in just one or two days. Finally, a first attempt at developing an aerobic analogue to the BMP test is undertaken. In theory, an aerobic method would complete much faster, without any loss in accuracy or precision.

1.6. Organization of the Thesis

This thesis is partitioned into six chapters. Chapter 2 provides a literature review of BMP methods relating to MSW, as well as background theory on gas production in waste and both the LOI and specific gravity procedures. Experimental procedures, materials, and equipment used are all described in Chapter 3. Chapter 4 presents the results from all experimental procedures. Detailed

analysis of those results is presented in Chapter 5. Finally, Chapter 6 highlights the conclusions that may be drawn from this study, and outlines several possibilities for further research based on this work.

Chapter 2 Background and Literature Review

2.1. Introduction

This chapter presents background information related to the use of the BMP test method on MSW. Included is an overview of the anaerobic biodegradation process as it occurs within waste, as well as a comprehensive review of the published literature relating to the BMP test, both as a general method, and more specifically its use on MSW. Also included is a review of the various factors that may influence the results of a BMP test. Lastly, a short description of some basic soil testing techniques that may be useful in attempting to improve the BMP test method are given.

2.2. Gas Production in MSW Landfills

The production of gases within full scale MSW landfills can be broken down into four distinct stages: I aerobic; II anaerobic non-methanogenic; III anaerobic methanogenic unsteady; and, IV anaerobic methanogenic steady (Farquhar and Rovers, 1973). A plot of typical landfill gas composition over time is given in Figure 2.1.

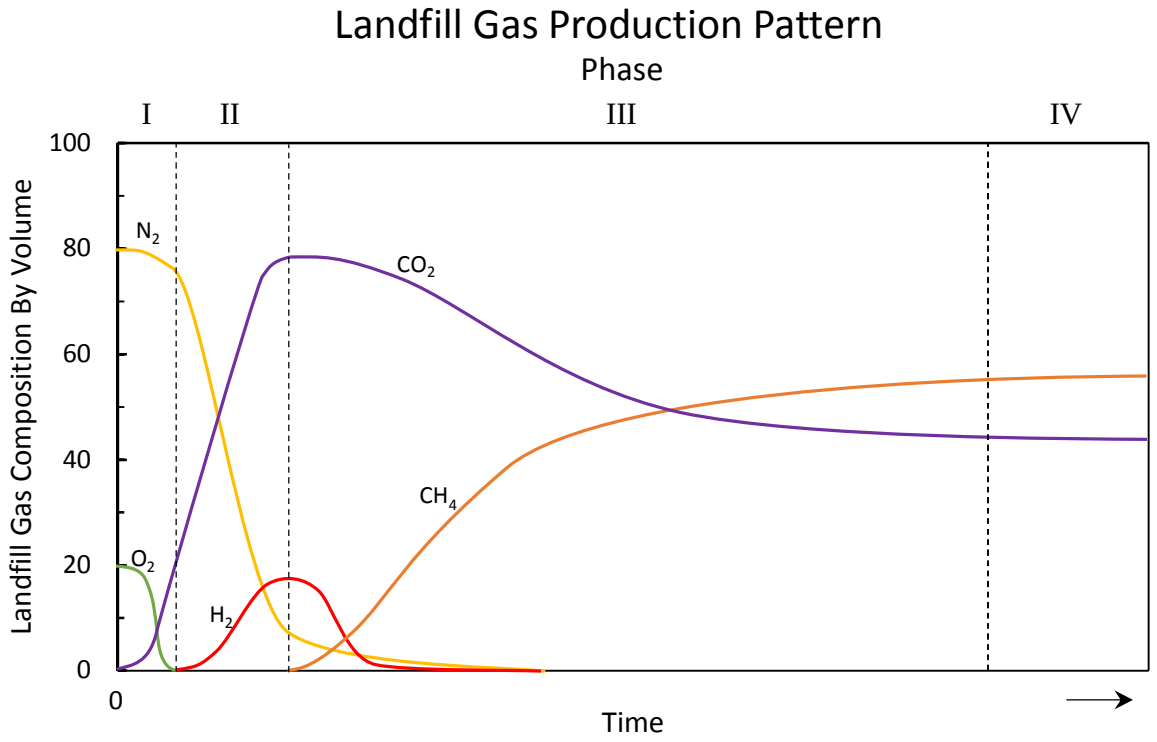


Figure 2.1: Typical landfill gas composition over time (reproduced from Farquhar and Rovers, 1973).

Stage I is the period wherein any oxygen present as the waste was placed is depleted in a brief period of aerobic activity. Stage II marks the beginning of anaerobic activity, but methanogenesis cannot yet occur, likely because there is still insufficient CO_2 in solution to act as a H_2 receptor (Farquhar and Rovers 1973). In Stage III methanogenesis begins and CH_4 concentrations slowly increase, while CO_2 concentrations decrease, until a steady state is reached, marking the beginning of Stage IV. The first two stages occur very rapidly and are important in terms of measuring the BMP only because they serve to delay the onset of methanogenesis. In an operating landfill Stage III begins within 2-3 months and the onset of Stage IV occurs within 5-7 months (Gregory and Browell, 2011). In a BMP test, anaerobic conditions are present from the beginning, and thus the duration of Stage I is negligible. The inclusion of an anaerobic inoculant serves to decrease the duration of Stages II and III, so that the effect of the brief duration of non-methanogenic activity is greatly reduced and the steady-state Stage IV is achieved much more rapidly.

2.3. Anaerobic Biodegradation Process in Waste

The progression of anaerobic biodegradation in waste is quite complex, with multiple processes occurring simultaneously at different relative rates. The complete, multistep process is nicely summarized in Figure 2.2.

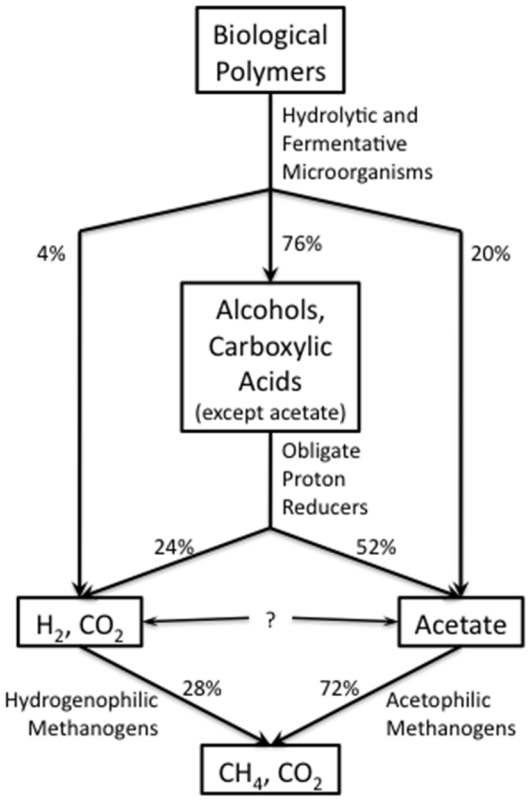


Figure 2.2: Complete, multistep methanogenic process, as it occurs within landfill systems (reproduced from Barlaz et al., 1990).

For simplicity, this process is often considered as four distinct steps: hydrolysis, acidogenesis, acetogenesis, and methanogensis (Francois et al., 2007). Each step is associated with its own group of microorganisms, all of which may be found within fresh waste (Barlaz et al. 1990). Hydrolysis and acidogenesis are governed by a diverse group of microorganisms known as hydrolytic and fermentative microorganisms (Barlaz et al. 1990). Hydrolysis is the breaking down of organic polymers such as carbohydrates, fats, and proteins into various sugars, amino acids, long-chain carboxylic acids and glycerol. Acidogenesis is the stage where these intermediate products are then further broken down into short-chain carboxylic acids, H_2, CO_2, and alcohols, with some acetate. Acetogenesis is the process by which acetogens convert the short-chain

carboxylic acids and alcohols into acetate, CO_2, and H_2. The final stage is the conversion of acetate and H_2 into CO_2 and methane by methanogenic microorganisms.

Although methanogenesis is a natural and common occurrence within landfill systems, on a smaller scale the process can actually be quite delicate. The majority of the pathways to produce methane in a landfill require that it be converted from acetate, and the production of methane from acetate by methanogenic bacteria is only barely energetically favourable (Barlaz et al. 1990). This explains why the production of methane within waste is such a slow process.

2.4. History of the BMP Test

2.4.1. General BMP

The BMP test was first described in 1979 by Owen et al. It was intended as a method to determine relative biodegradability of various organic substrates, as well as a batch method for evaluating the possible inhibitory effects of various substances on the anaerobic biodegradation process. The method described uses small, 250 mL reagent bottles, containing less than 2 g/L of degradable material with approximately 160 mL of carefully prepared nutrient media, inoculated with BMP seed (typically sludge from an anaerobic digester) just prior to transfer. Gas accumulates within the sealed bottles and is removed periodically using a glass syringe by gently twisting the plunger until equilibrium is achieved between the reagent bottle and atmospheric pressure. Since its publication, the BMP test method described by Owen et al. (1979) has been modified and used by many researchers, in several areas of study, though no generalized standard method exists.

There are several ASTM standards relating to the biodegradability of plastics, which are similar to the BMP test, such as ASTM D5210 and D5526, though these are too specific to be applied to a more general medium. The ASTM standard most relevant to BMP, E2170, described the “anaerobic biodegradation potential of organic chemicals under methanogenic conditions”, but was withdrawn in 2013, with no replacement, due to its “limited use by industry”. A review of the published literature reveals that though commonly referenced in other works, these standards have rarely been put to use by researchers.

The VDI 4630 (2006) is a standard published by the Association of German Engineers for the fermentation of small amounts of organic materials. A review of the published literature did not reveal any researchers using this method for the digestion of MSW.

2.4.2. MSW Specific BMP

The earliest example of the BMP test being used on unprocessed MSW is described by Owens and Chynoweth, 1993. They used a sample mass of MSW that contained 2 g of volatile solids (VS) per 1 L of sample solution. In their 100 mL total solution, this amounted to a mass of MSW containing a mere 0.2 g of VS, which is likely less than 1 g of total dry MSW. They specified a minimum incubation time of 60 days at 35°C, with the final reading from their tests being taken after approximately 75 days. Gas was collected for analysis by periodically inserting a syringe through a rubber septum in the vessel's cap, and allowing equilibration to ambient barometric pressure.

All subsequent publications of BMP methods used on MSW appear to be traceable back to the paper by Owens and Chynoweth, with slight to moderate modifications in terms of incubation times, amount of material digested, etc. Table 2.1 presents a variety of test methods that have been used by various researchers to evaluate the BMP of unprocessed MSW.

The methods of gas collection also differ between researchers. Many subscribe to the method put forth by Owens and Chynoweth, using a syringe and allowing equilibration to ambient pressure. For those using much greater sample masses however, the quantities of gas being produced are too great for this method to be practical. The majority of these researchers use a water displacement column to measure their gas volumes. In all cases, the most common method of analyzing gas composition is a gas chromatograph.

Currently, there are a few agreed upon methods for determining the BMP of MSW by specific agencies, but acceptance of these methods as standards does not commonly extend across international borders. The BM_{100} test, as described by Godley et al. 2007 and Bockreis et al. 2007 (see Table 2.1), is adapted from a sewage sludge digestion method, and is widely used in England and Wales. The method described by Binner and Zach, 1999; Bockreis et al. 2007; and Heerenklage and Stegmann, 2005 is a German and Austrian standard known as the GB_{21}. Heerenklage and Stegmann also describe another German/Austrian standard utilizing a much larger sample known as the GS_{21}.

Table 2.1: Summary of BMP test methods used on MSW.

Researcher & Year	Test Name	Incubation Time (days)	Mass of Sample (g)	Total Volume of Liquids and Solids(mL)	Incubation Temp. (°C)
Owens and Chynoweth, 1993	Not Specified	75	<1	100	35
Binner and Zach, 1999; Heerenklage and Stegmann, 2005; Bockreis et al. 2007	GB_{21}	21	50	350	35
Jokela et al. 2002	Not Specified	110	<30	1600	35
Hansen et al. 2004	Not Specified	50	10	500	55
Heerenklage and Stegmann, 2005	GS_{21}	21	800-1500	Not specified	40
Kelly et al. 2006	Not Specified	45	2	100	35
Francois et al. 2007	Not Specified	30-60	30	~700	35
Godley et al. 2007; Bockreis et al. 2007	BM_{100}	100	20 g Volatile Solids	Not specified	35
Ivanova et al. 2008	Not Specified	Unlimited	100	700	30
Zhu et al. 2009	Not Specified	20	2.65	500	50
Bayard et al. 2010	Not Specified	90	10	1100	35
Bao, 2011	Not Specified	90-Unlimited	Not Specified	95	37
Chantou et al. 2011	Not Specified	21 and 55	3.2	Not Specified	35

2.5. Factors Affecting BMP

As a measurement relying on biological processes, performing the BMP test accurately in a laboratory environment can be very tricky. The anaerobic nature of the test also creates unique challenges that must be addressed. The following is a list of factors that can affect the outcome and/or accuracy of the BMP test.

2.5.1. Oxygen Availability

As explained above (Section 2.2), the production of methane in waste requires anaerobic conditions. Any infiltration of oxygen will quickly be consumed and result in greater CO_2 production and less CH_4 production for a brief period. In a small-scale laboratory setting, this brief period of increased CO_2 production could significantly skew the results. Therefore, anaerobic conditions must be strictly maintained from the beginning of the test.

2.5.2. Nutrient availability

Biological organisms require many different things to function and live. Although there may be plenty of organic compounds available as food, if there are insufficient levels of nitrogen, phosphorous and potassium, biological activity will cease (Barlaz et al. 1990). In a sample of MSW these nutrients may or may not already be present. Happily, the anaerobic sewage sludge used by many researchers as a source of microorganisms also provides a sufficient source of nutrients for those microorganisms (Barlaz et al. 1990). Dong et al. (2013) showed that phosphorous is often the limiting nutrient in regards to the stabilization of solid waste in an anaerobic landfill.

2.5.3. Chemical Inhibition

Just as certain chemicals and nutrients are required for anaerobic microorganisms to survive, an overabundance of some chemicals or nutrients can have the opposite effect. In anaerobic conditions, ammonia (NH_3) and the ammonium ion (NH_4^+) are the most common causes of chemical inhibition (Yenigün and Demirel, 2013). Other causes of chemical inhibition may be excess concentrations of sulfide, light metals (Na, K, Mg, Ca, and Al), heavy metals (particularly Cr, Fe, Co, Cu, Zn, Cd, and Ni), and a wide variety of organic compounds. There are many

possible mechanisms for inhibition, depending greatly on the source or chemical that is causing it (Chen et al. 2008). Other factors affecting the mechanism of inhibition are temperature, pH levels, the nature of the material being digested, the characteristics of the microbiological community, etc. (Yenigün and Demirel, 2013).

2.5.4. Physical Inhibition

In wood products, a portion of the highly degradable cellulose is physically associated with non-degradable lignin, effectively shielding the cellulose from microbial attack. The milling process dissociates some of the cellulose from the lignin, resulting in an increased amount of microbially available cellulose, and therefore increased BMP values (Ivanova et al. 2008; Stinson and Ham, 1995). However, if waste samples are to be representative, while remaining at a manageable size, this effect is unavoidable. A balance must be struck between keeping sample sizes small enough that testing is simple and repeatable, and not grinding the material so fine that the samples are no longer representative.

2.5.5. pH levels

The microbiological organisms involved with methanogenesis are sensitive to pH levels. The pH value at which methanogenic bacteria remain most active varies with waste composition and BMP test method (Fantozzi and Buratti, 2011). Dong et al. (2013) stated that the optimum pH is nearly neutral, and usually in the range of 6.5 to 8.2. Under mesophilic conditions, the optimum pH range has been shown to be 6.8 to 7.4 (Barlaz et al. 1990; Liu et al. 2008). One of the many ways in which methanogenesis can be inhibited within waste is when the activity of the hydrolytic and fermentative organisms exceed that of the acetogens and methanogens, causing acidification through a buildup of H_2 and carboxylic acids, reducing the pH to levels below what the methanogens can handle (Barlaz et al. 1990). Liu et al. (2008) showed that methanogensis is severely inhibited at pH conditions less than approximately 6.0.

2.5.6. Temperature

Temperature also has a significant impact on the activity of the microbiological organisms within landfills. Mata-Alvarez and Martinex-Viturtia (1986) found that the peak methane production rate occurs at 42°C, but the maximum cumulative methane production occurs between 34°C and 38°C.

Anaerobic digestion is an overall exothermic process, and the interior of a full-scale landfill often exceeds 40°C (Farquhar and Rovers, 1973), therefore it is sound practice for researchers to conduct BMP testing at peak methane production temperatures.

2.5.7. Leakage

Information on the effect of leakage on BMP test methods is difficult to come by. One study of small-scale household biogas digesters suggested that leakage rates may be as high as 10% of the total biogas produced on average, and as high as 3.1% even in well-maintained digesters (Bruun et al. 2014). In a laboratory setting, the amount of leakage is likely to be much less, but clearly, any system involving pressurized gases has the potential to develop leaks, and this must be managed as well as possible.

2.5.8. CO_2 Solubility

It is a common practice for researchers to measure the volume of gas production using a water displacement column (Raposo et al. 2011; Reddy et al. 2011; Stinson and Ham, 1995; etc.). CO_2 is known to be relatively soluble in water, so this may result in measurements of gas volumes and CO_2 concentrations that are below their actual values. Accurate gas concentrations can be assured by taking measurements before or during gas transfer from the vessels to the water column. Dissolution of CO_2 is not instantaneous, but follows a first order kinetic model (Mojtaba et al. 2014), therefore the effect of CO_2 dissolution can be reduced by taking volume readings as quickly as possible. The amount of CO_2 dissolved may also be reduced by adding salt to the water (Liu et al. 2011).

2.5.9. Poor Reproducibility

Reproducibility (measured by the coefficient of variation) is difficult to achieve in a BMP test. Two interlaboratory studies on BMP testing are known to have been conducted; one by Pagga and Beimborn (1993), and the other by Raposo et al. (2011). Without accounting for outliers, the two studies found average coefficients of variation for BMP testing of 29% and 24%, respectively. Excluding outliers, the average coefficients of variation were reduced to 18% and 9%, respectively. These large coefficients of variation, combined with the fact that each data set

contained several significant outliers, indicate that large variations and low reproducibility in BMP testing are not uncommon.

2.6. Aerobic Methods

In the presence of oxygen, waste degradation will proceed aerobically. Rather than CH_4 and CO_2, the main products of aerobic degradation are CO_2 and water. Aerobic conditions do not usually occur naturally in landfills, but there are several methods by which human intervention can create primarily aerobic conditions within a landfill (Ritzkowski and Stegmann, 2012). Aerobic decomposition is a much quicker process than anaerobic decomposition. This makes aerobic tests very desirable for researchers, because they can be carried out in just a few days, rather than weeks or months. Several aerobic tests exist for the determination of waste stability. Some of them are:

- Activity Test (AT_4 or RI_4) (Binner and Zach, 1999; Heerenklage and Stegmann, 2005; Cossu and Raga, 2008);
- Dynamic Respiration Index (DRI) (Scaglia et al. 2010); and,
- Specific Oxygen Uptake Rate (SOUR) (Lasaridi and Stentiford, 1998; Scaglia et al. 2007).

However, these methods are only used to measure the rate of gas production, as an indication of the waste reactivity; they do not provide an indication of total gas generating potential. Scaglia et al. (2010) proposed a relationship between the Dynamic Respiration Index and BMP (what they called the Anaerobic Biogas Potential), but as yet, no aerobic test directly analogous to the BMP has been developed.

2.7. Loss on Ignition

The ASTM defines loss on ignition (LOI) as "the loss in mass of the test specimen when heated under controlled conditions of temperature, time, atmosphere, specimen mass, and equipment specifications" (ASTM D7348). For waste research, the temperature that LOI is most commonly measured at is approximately 550°C. It is generally accepted that at this temperature all organic compounds will burn off, leaving only inorganic residue behind. In the literature, terms like total organic matter (OM) (Meissl and Smidt, 2007) and volatile solids (VS) (Arias et al. 2012; Kumar and Goel, 2009; Scaglia et al. 2010; etc.) are used synonymously with LOI at 550°C.

If the LOI at 550°C is truly a measure of the organic content of a sample, then it follows that it may serve as an approximate indication of the remaining methane potential. However, there

are several disadvantages to using the LOI as an indicator of waste stability. First, any volatile inorganic substances such as nitrogen oxides, ammonia, and hydrogen sulfide will be included in the measurement; and second, a portion of the organic fraction will be composed of biologically inert material such as plastics, which do not contribute to BMP (Soyez and Plickert, 2002). It is probable though, that some portions of the organics will volatilize at temperatures below 550°C. In 2011, Gebert et. al investigated the relationship of total organic carbon (which they also found had a close relationship to LOI) and BMP, but found no significant correlation. However, their research did not attempt to account for non-biodegradable organics, nor did it investigate LOI at varying temperatures. In fact, no research was found investigating the relationship between BMP values and LOI values at various temperatures. It is possible that some better relationship between BMP and LOI may exist at a different temperature, or even through some manipulation of LOI values at multiple temperatures.

2.8. Specific Gravity

The Specific gravity of a substance is a unitless measurement of the density of the substance, relative to the density of distilled water at 4°C. Measuring the specific gravity of soil particles is a common practice in geotechnical applications. There exist at least two ASTM standards for the determination of the specific gravity of solid soil particles. The first (ASTM D854) relies on principles of water displacement, and the second (ASTM D5550) relies on principles of gas displacement. ASTM D5550 requires specialized equipment capable of precise gas pressure and volume measurements, but has the advantage of being able to provide accurate measurements for samples which may contain solids that readily dissolve or float in water.

The degradable fraction of MSW is mostly composed of relatively low-density organic material. As degradation progresses, these low-density solids are consumed and converted into gas. Because of this, the specific gravity of MSW should theoretically increase over time, and may be considered as an indicator of the carbon decay of waste (Drut et al. 2011). If the specific gravity serves as an indicator of carbon decay, then it is possible that there may be a direct correlation to the remaining BMP. Another possibility is that the specific gravity may be used to quantitatively identify outliers, thereby improving an LOI-BMP correlation.

2.9. Summary

The BMP test method has been used to evaluate the methane potential of MSW for approximately 20 years. In recent years, the number of published articles related to the BMP of MSW has spiked dramatically. Despite all this interest in the subject, the published literature still does not agree on the best method for performing the BMP test on MSW. There is great variation in the incubation time, mass of sample, and the total volume of the mixture of liquids and solids, as well as gas collection and analysis methods.

The BMP test is fairly complex, and there are many factors that can adversely affect its results. This thesis will attempt to address all of these issues and present an alternative method for performing the BMP test on MSW. One of the most undesirable traits of the BMP test is its lengthy time requirement. This thesis will also attempt to provide an alternate method of estimating BMP by correlating BMP results to quicker and simpler tests.

Chapter 3 Materials and Laboratory Testing Program

3.1. Introduction

This chapter presents details about the various materials and equipment used in this study. It also describes the testing procedures that were used to carry out the research. Waste materials for this study came from four individual sources. Test procedures included BMP, sequential loss on ignition (LOI), specific gravity, and an attempt at an aerobic BMP analogue dubbed the Biochemical CO_2 Potential (BCP).

3.2. Materials Used

Materials for this study were collected from four individual sources, as shown in Table 3.1.

Table 3.1: Sources of waste material for this study.

Waste Source	Waste Type	Number of Samples
Rural Municipality of Wood Buffalo (RMWB) Landfill, Fort McMurray, AB	Mixed MSW	20
Spadina Landfill, Saskatoon, SK	Mixed MSW	10
Barrie, ON	Processed Waste (Particles < 51 mm only)	4
Industrial Waste Stream, Cowlitz County, WA	Variety of Fresh Industrial Waste	5

The materials from RMWB, Barrie, and Cowlitz County, were sourced from various industry contacts who had expressed a need for BMP testing. The materials from the Spadina landfill in Saskatoon were gathered from the leftover materials of a separate waste research project being conducted at the U of S at the same time as this study.

3.2.1. Rural Municipality of Wood Buffalo Samples

Cores from the RMWB landfill were drilled using a 0.108 m diameter sonic drill rig. Five boreholes were drilled in each of four separate landfill cells that varied in age and depth for a total of twenty cores. Each core was drilled to the presumed depth of the base of the landfill (exact depths were unknown) and ranged in length from 7.0 m to 13.7 m. Sample recovery was very good; sometimes exceeding 100% due to expansion of the cores. Cores varied widely in age, composition, water content, degree of compaction, and state of decomposition. Following collection, each core was bagged, sealed, and transported to the lab at the U of S where they were kept in a climate controlled room at 4°C until they could be processed.

3.2.2. Spadina Samples

Cores from the Spadina landfill in Saskatoon were drilled using the same sonic drill rig that had been used at RMWB. Further material was collected from near surface, using a backhoe. Whether cored or excavated, each sample was bagged, sealed and transported to the U of S where they were kept in a climate controlled room at 4°C until they could be processed.

3.2.3. Barrie Samples

The samples from Barrie, Ontario were sent to the U of S from a waste processing operation attempting to separate large amounts of sand from the landfill. The waste was processed through a 51 mm screen and samples were collected from material passing the screen. Four samples were collected on four different days. Each sample was one 5-gallon pail of material. The pails were then sealed, and shipped to the U of S where they were kept in a climate controlled room at 4°C until they could be processed.

3.2.4. Cowlitz Samples

The Cowlitz county samples were not what would be considered “classic” MSW. They were intercepted from three industrial waste streams going to the landfill. The reason for including these samples was to evaluate the test methods described in this research on a broader range of waste materials. The three samples sent were deink sludge and wastewater treatment solids from a pulp/paper mill, and autoshredder fluff. Each sample was shipped to the U of S in an individually sealed 5-gallon pail, where they were kept in a climate controlled room at 4°C until they could be processed.

3.2.5. Anaerobic Inoculum

Anaerobic sewage sludge was collected periodically from the Saskatoon Wastewater Treatment Plant for use as BMP inoculant. The sludge was collected as needed, from whichever of the three digesters was active at the time of collection. Sludge was collected and transported in 1 L, sealed plastic bottles to maintain its anaerobic nature. The bottles were stored in a climate controlled room at 4°C until they were needed, usually only 1 to 2 days.

3.2.6. Aerobic Inoculum

An aerobic inoculum was required for the BCP test. BOD seed is a collection of biological cultures specialized for aerobic degradation that is most often used for biochemical oxygen demand testing. The BOD seed used in this study came in dehydrated pill form. The cultures had to be activated by introducing them to water, and aerating them for several minutes.

3.3. Scope of the Testing Program

BMP and sequential LOI tests were both carried out on each of the waste samples tested. Specific gravity testing was carried out on all samples; excluding those from Barrie, ON; which were few in number and had little variation between them.

Certain aspects of the BMP test were varied between individual test batches. This was to evaluate the relative impacts, if any, of these aspects on the BMP testing process. Ultimate BMP value was not considered to be the only potential impact; properties such as gas production rate and length of the initial lag phase were also considered.

The BCP was not performed as a full-scale study, but merely a preliminary test of concept. It was only attempted on 3 samples, and is intended to provide a possible starting point for expanding on this study.

Two of the four sample sources for this study provided mixed MSW sourced from borings taken through landfilled waste. Of the other two sources, one provided a processed landfill material; and the other provided several types of fresh industrial waste, intercepted before reaching the landfill. These last two sample sources provided the means to evaluate the efficacy of these BMP methods on a broader range of waste materials.

3.4. Sample Processing

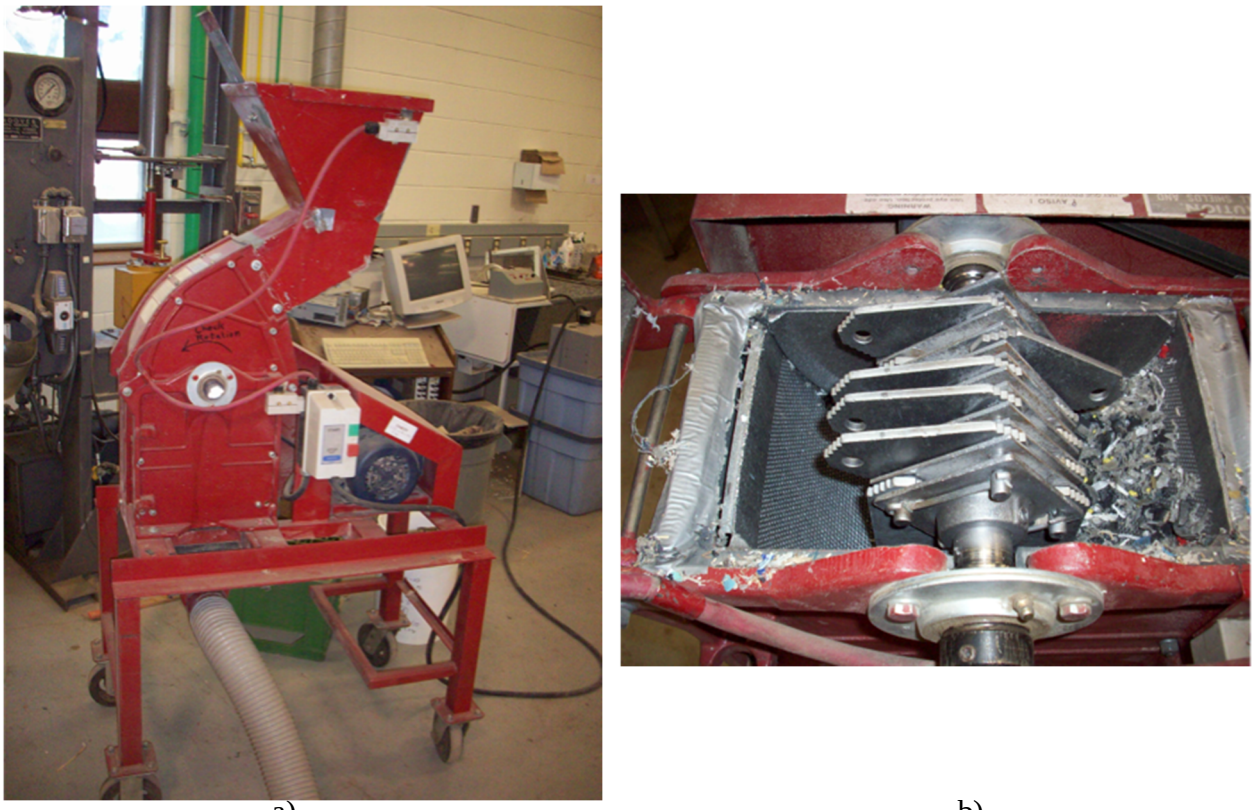
3.4.1. RMWB Samples

Once in the lab, each core was laid out in sequence, and reduced by arbitrarily collecting the first 0.3 m of every 0.9 m. For the longer cores, the first 0.3 m of every 1.2 m was collected, to keep samples at a manageable size. Two typical cores are shown in Figure 3.1.

a) b)

Figure 3.1: Typical waste core photo logs showing a) BH7 and b) BH19.

Once reduced, each sample was mixed thoroughly by hand and divided into two replicate subsamples ('A' and 'B') using the technique of "quartering" (Ballinger and Shugar, 2011). The two replicate samples were then weighed and dried in bulk in a large geotechnical oven at 60°C. The bulk drying process resulted in a small amount of residual water remaining in the samples. This residual water was measured and corrected for later on, using data from the earliest stages of the sequential LOI testing. Following the bulk drying, subsample 'B' was set aside as extra material. Subsample 'A' was subsequently used for all testing, but was first further processed by shredding to a particle diameter of approximately 3 mm, using the hammer mill shown in Figure 3.2. Figure 3.3 shows a typical waste sample before and after shredding.



a) b)

Figure 3.2: a) Hammer mill exterior, showing feeding chute and suction hose. b) Hammer mill interior, showing pivoting hammers and 3 mm screen.

a) b)

Figure 3.3: a) Typical waste sample before milling. b) Typical waste sample after milling.

3.4.2. Spadina Samples

The goal with these samples was not to characterize the landfill as a whole, but to collect samples that would provide a wide range of BMP values for research purposes. A broad range of BMP values was desired so that possible correlations to simpler and/or quicker tests could be fully explored. Thus, a few samples were created randomly from cores, using the method described in Section 3.4.1, but most were created by selecting material rich in various components such as paper, plastic, wood, and soil. The material was selected by subjective visual observation only, as the goal was to extend the range of BMP values in the samples, not to characterize specific waste components. The main components of each sample are listed in Table 3.2. Each sample was reduced to a maximum particle diameter of 3 mm using the hammer mill.

Table 3.2: Main components of Spadina waste samples, as determined visually during collection.

Sample	Main Component(s)
S1	Wood & Paper Products
S2	Soil & Degraded Material
S3	Paper Products
S4	Wood & Paper Products
S5	Random (Mostly Soil)
S6	Random
S7	Random
S8	Random
S9	Plastics
S10	Wood

3.4.3. Barrie Samples

Due to the nature of the material (being the less than 51 mm particles collected from a landfill mining project), theses samples mostly consisted of sand and required very little further processing aside from drying at 60°C. What few particles that did require further reduction were broken apart by hand, or cut into smaller pieces using scissors.

3.4.4. Cowlitz Samples

These samples were each tested independently, as well as in two combined mixtures. The first mixture was a combination of all three samples; the second was a combination of all three samples mixed with a more typical MSW material. Sample BH16 from RMWB was selected as the MSW for this mixture, based on its fairly average and consistent BMP values, as determined from previous testing. The components of the five samples are given in Table 3.3.

Table 3.3: Components of Cowlitz waste samples. Percentages of mixed samples are on a wet weight basis.

Sample	Components
D	Deink Sludge
W	Wastewater Treatment Solids
A	Autoshredder Fluff
CS	Combined Sample (40% D, 36% W, 24% A)
CS/MSW	Combined Sample with MSW (33% CS, 67% MSW)

As received, the particles of the deink sludge and the wastewater treatment solids were fine enough that no further shredding was required. The precise chemical composition of these materials was unknown, and it was feared that some of the components of these samples may volatilize at very low temperatures; even less than 60°C. Therefore, these samples were air dried on plastic sheets. The autoshredder fluff was mostly large pieces of foams and hard plastics that was air dried and subsequently shredded to a maximum particle diameter of 3 mm using the hammer mill.

3.5. BMP Equipment

3.5.1. Test Vessels

Rather than use the small 160 mL serum bottles of some other methods (Bao, 2011), this method used 2 L glass bottles as the test vessels. This allowed the test to be performed on a much larger volume of waste; up to 200 times larger compared to some methods. Each vessel was fitted with two openings: the first opening was attached to a 1 L Tedlar® gas sampling bag; the second opening was fitted with a short piece of tubing and a hose clamp, for removal and collection of excess gas. The use of gas sampling bags over the rubber septum and syringe method helped to save space in the lab. With up to 200 times more waste in a sample there was an equal increase in the volume of gas produced. The gas sampling bags provided a useful visual indication of gas production, allowing them to be released and monitored repeatedly as needed, without ever allowing the vessels to over pressurize. To increase the mass of sample using the rubber septum and syringe method (where only one gas measurement is taken) would require a vessel large to contain the increased volume of gas, or thick enough to withstand the over-pressurization. Similarly, the size of the syringe used for sampling and measuring the gas volume would need to increase proportionally. This would very quickly become impractical. Figure 3.4 shows several active BMP test vessels arranged in the incubator.

Figure 3.4: Active modified BMP test vessels in the incubator.

3.5.2. GEM™2000

Gas composition analysis was performed using a GEM™2000 landfill gas monitor (Landtec Inc., Calif., USA). The GEM measures CO_2 and CH_4 composition using dual wavelength infrared cells, and O_2 composition using an internal electrochemical cell. It was designed as a field instrument specifically for monitoring composition and flow of gases from landfill gas extraction systems. This means it has several disadvantages over gas chromatographs, and other lab-based gas composition instruments:

- It is restricted to measurement of only CH_4, CO_2, and O_2;
- It requires a minimum volume of about 300 mL of gas before it can get a stable reading; and,
- At gas compositions of 15% and above its typical precision, as listed in the user's manual, is only ±3.0% for measurement of CH_4 and CO_2, and ±1.0% for measurement of O_2 (Landtec, 2009).

The first disadvantage is not an issue for BMP testing since CH_4 is the only gas of real interest (though, monitoring of CO_2 and O_2 is useful in detecting leaks, or inhibitory effects during testing).

The other two disadvantages can be addressed by taking frequent measurements of large quantities of gas. The hypothesis is that any errors introduced due to the lack of precision of the instrument readings should be compensated for if many measurements are taken using large volumes of gas for each measurement. A big advantage of the GEM is that it has an internal pump. If the exhaust tube is fed directly into the water column, this means that the GEM can be used to transfer gas from the BMP vessels to the water column while measuring gas concentrations. In this arrangement, gas concentrations are measured before any CO_2 is lost to dissolution. Other advantages of the GEM for BMP testing are its affordability, its simplistic operation, and its portability. All of which make it a much more accessible option for anyone wishing to carry out BMP tests; particularly under field conditions.

The GEM requires calibration in order to maintain accuracy for gas composition readings. A two point calibration method is used for each of the three gases measured, using standard gas mixtures available from Landtec Inc. Calibration was conducted by connecting the gas cylinders directly to the GEM through a small length of tubing. The exact composition of the gas being used was then input, and the GEM turned on. Gas was allowed to flow until the composition readings had stabilized, then the readings adjusted to match the known composition of the calibration gas. The first gas mixture used consisted of 50% methane, 35% CO_2, and a balance of nitrogen. This gas mixture was also used to calibrate the O_2 sensor at 0%. The second gas mixture used consisted of 4% oxygen and a balance of O_2. This gas mixture was also used to calibrate the methane and CO_2 sensors at 0%. Calibration was carried out approximately once per week.

3.5.3. Other equipment

Other equipment included an upright, fridge-sized incubator, with a capacity to hold 42 of the 2 L vessels used for testing; and a water displacement column crafted from a section of 51 mm diameter clear plastic tube. The total volume of the tube was slightly over 1300 mL.

3.6. BMP Procedure

The BMP test method used was modified from Owens and Chynoweth, 1993. The main modification was a large increase in the mass of sample tested. It is hypothesized that a larger sample will allow for much greater accuracy in the determination of the BMP, since a large sample is much more likely to be representative of the waste. If 10 g of sample from a 2 kg parent sample

of homogenized, shredded waste are measured for BMP, the confidence interval for that sample being within 95% of the actual BMP is only 30.9%. If 200 g of sample are taken from the same 2 kg parent sample, the confidence interval is reduced to 6.58% at a confidence level of 95%. The required minimum accuracy of the test is, of course, dependent on the project it is used for, but clearly, greater accuracy is always desirable.

The other major change to the Owens and Chynoweth method is that gas compositions will be measured repeatedly over time using the GEM, rather than only once using a GC. It is expected that the larger sample size will also help to make up for the loss in precision that comes with using the GEM over a GC. This is shown in the following example:

Assume a sample of waste produces 50 mL of landfill gas per gram at a ratio of 60% methane to 40% CO_2. If a 2 g sample is tested, and the precisions of the instruments are as follows:

- Mass precision, $P_{mass} = \pm 0.01$ g
- Gas concentration precision $P_{conc} = \pm 0.01\%$
- Gas volume precision, $P_{vol} = \pm 0.1$ mL

Then the possible range of BMP calculated is 29.82 mL/g to 30.19 mL/g.

Now assume a 200 g sample of the same waste is measured using instruments with 10 times less precision:

- $P_{mass} = \pm 0.1$ g
- $P_{conc} = \pm 0.1\%$
- $P_{vol} = \pm 1$ mL

The possible range of BMP calculated in this scenario is 29.93 mL/g to 30.07 mL/g.

It is clearly shown that in the second scenario (using a larger sample with less precise instrumentation) the possible range of calculated BMP values is narrower than the first scenario, indicating increased precision overall. On top of that, this example doesn't take into account the effect of taking multiple measurements over time. By taking multiple measurements, the distribution of both positive and negative precision errors should cancel each other out to some degree once a fitted curve is applied to the data as a whole. This means that by combining multiple measurements, the overall precision should increase even more. It is important to note however,

that this assumes the measurement errors all follow a normal probability distribution and are not subject to a systematic bias.

3.6.1. Test Set-Up

Triplicate vessels were set-up for each sample according to the following procedure:

1. Approximately 200 g of shredded MSW was placed into the vessel, and the exact mass recorded. In some cases, excessive "fluffiness" of the shredded sample, typically associated with a high content of fibrous material (eg. wood and paper), meant that getting 200 g into the vessel was impractical. In these cases, the mass of sample added was reduced as necessary.
2. Between 100 and 200 mL of anaerobic sewage sludge was added as an inoculum. The volume was different between some test batches to study the effect(s), if any, of the amount of inoculum.
3. Anywhere from 300 to 1500 mL of distilled, de-aired water was added to provide a wet, oxygen-free environment for the promotion of anaerobic microbial activity. Again, the volume was different between some test batches to study any effects the volume of water might have. Within particular test batches, certain samples required additional water due to their excessive "fluffiness".
4. The vessel was then swirled until the waste, water, and inoculum were thoroughly mixed. The swirling action was preferred to avoid any infiltration of air into the mixture.
5. As quickly as possible, the vessel was capped, and purged with either nitrogen or argon gas for 1 minute, to remove as much air as possible. While purging, the gas sampling bag was removed so gas might flow into one port and out the other.
6. Immediately after purging, the fully deflated gas sample bag was attached and the hose clamp closed to seal the vessel.
7. As a precautionary measure, silicone was then applied to the area around the sample bag's stem. Initial tests showed that the bags used were prone to develop leaks in this area.
8. Lastly, the sealed vessel was placed inside an incubator at 35°C.

For every six bottles (two triplicate sets) a separate vessel was created following the same steps, minus the addition of waste material. This "blank" was then monitored in the same manner as the

regular vessels, to measure and account for any additional methane potential attributable to the inoculum. The cumulative methane potential of the blank was subtracted from the cumulative methane potential of each of its six corresponding test vessels after every reading.

3.6.2. Test Monitoring

Vessels were monitored initially every 24 hours, with decreasing frequency as the test proceeded and gas production rates slowed. If sufficient gas had been produced (greater than ~300 mL) the vessel was removed from the incubator and the clamped end attached to the GEM's sampling port. Once the clamp was open and the GEM turned on, the machine simultaneously pumped excess gas from the vessel, directly to a water displacement column, while measuring its composition in line. The process was carefully monitored, and the pump deactivated, just as the bag was deflated. Some simple calculations (shown in Appendix A) using Henry's law and the Ideal Gas law show that if the volume of gas being measured is small compared to the volume of the column, much more CO_2 is potentially dissolved in the water. For this reason, the volume of the water column chosen was just slightly larger than the capacity of the gas collection bags. The effect of CO_2 dissolution was also minimized by taking measurements as quickly as possible, since dissolution of CO_2 is not instantaneous. The addition of salt to the water column was not considered to be warranted, due to the precautions already taken, and the small potential for CO_2 dissolution that was calculated. Gas volume and composition were then recorded, and the vessel returned to the incubator. A schematic of the test monitoring setup is presented in Figure 3.5.

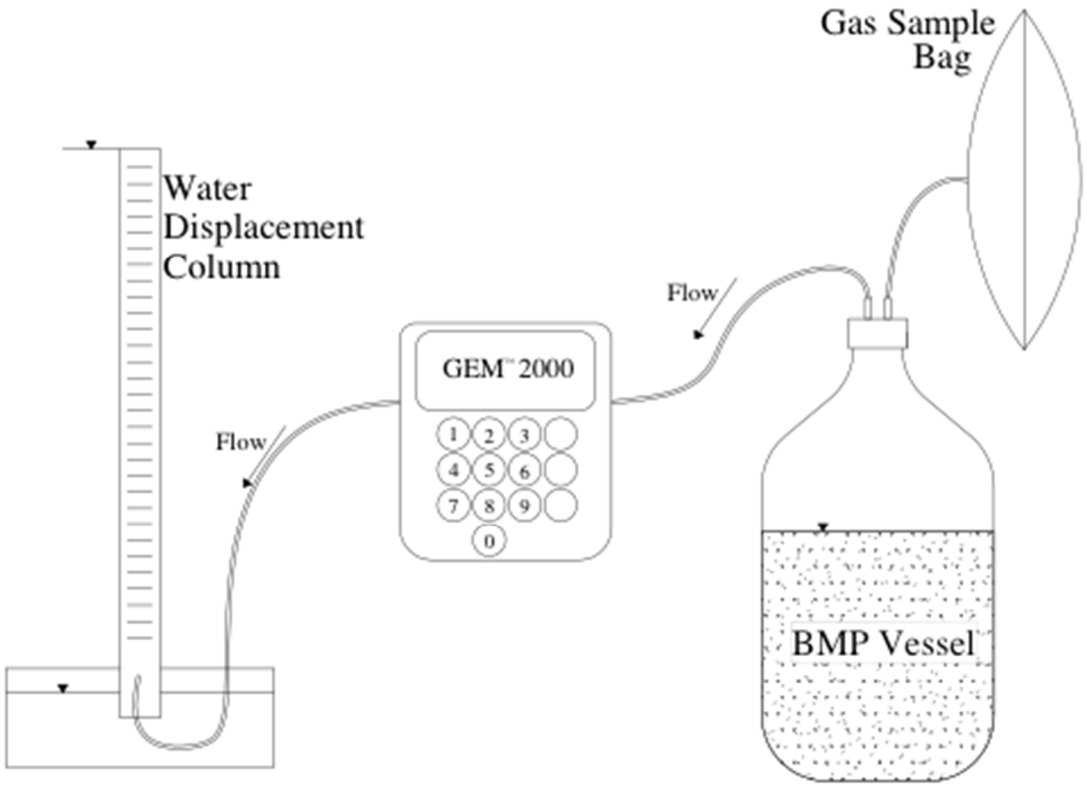


Figure 3.5: Schematic of test monitoring setup (not to scale). Gas is drawn from sample vessel using the internal pump of the GEM™2000, and composition is measured in-line before being exhausted to the water column.

The cumulative methane potential was calculated as the test proceeded by the following equation:

$$\boldsymbol{BMP_T = \frac{\left(V_H G_T + \sum_{t=0}^{t=T} V_t G_t\right) - \left(V_{BH} G_{BT} + \sum_{t=0}^{t=T} V_{Bt} G_{Bt}\right)}{M}} \qquad \text{(Eq. 3.1)}$$

Where: BMP_T = the biochemical methane potential at time, T; V_H = the headspace volume of the vessel; G_T = the fraction of methane gas recorded in the vessel at time, T; V_t = the volume of gas removed from the sample bag at time, t; G_t = the fraction of methane gas recorded in the vessel at time, t; subscript B indicates the same variables with respect to the corresponding blank vessel; M = the mass of the sample.

Monitoring of each vessel continued until the plot of cumulative methane potential over time showed a sufficiently horizontal curve to be considered the ultimate BMP. Figure 3.6: Anticipated shape of BMP curve. After a brief lag time, gas production follows a first order decay

model which asymptotically approaches an ultimate BMP value.Figure 3.6 shows the anticipated shape of the BMP curve.

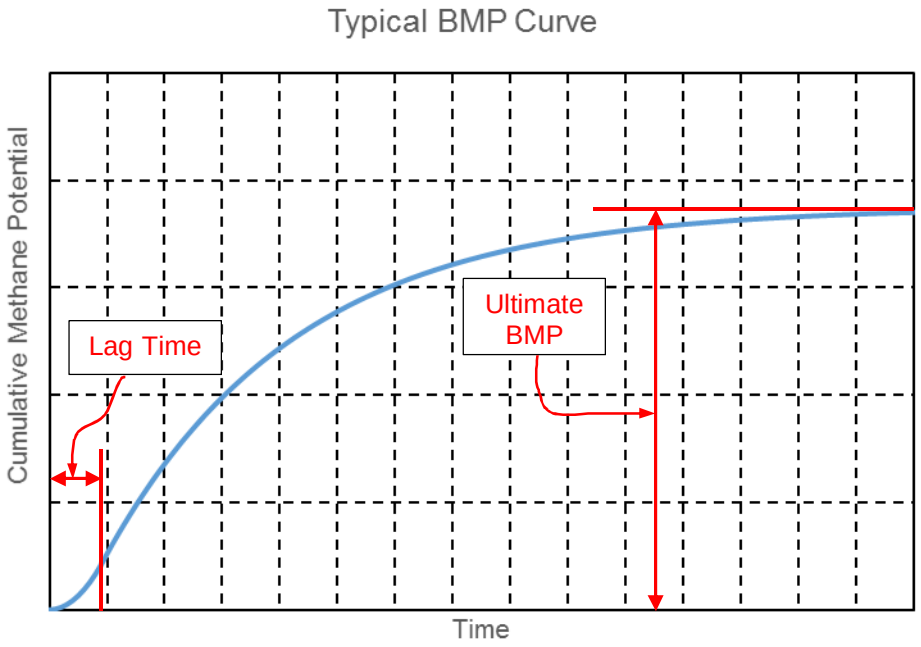


Figure 3.6: Anticipated shape of BMP curve. After a brief lag time, gas production follows a first order decay model which asymptotically approaches an ultimate BMP value.

3.7. Sequential LOI Procedure

There has been at least one attempt at correlating BMP with LOI in the past (Gebert et al. 2011), but that research did not attempt to account for either non-organic volatiles or non-biodegradable organics, nor did it consider LOI at more than one temperature. By performing LOI tests multiple times on the same sample at sequentially increasing temperatures, it was hypothesized that the different ignition temperatures of different substances would allow for a better accounting of the composition of a sample, and possibly a more statistically significant correlation.

For each individual sample, three large crucibles of known mass were filled with as much shredded waste as could reasonably fit in them. Dependent on the nature of the waste, this was anywhere between 20 and 150 g. Each crucible was then sequentially heated to 60, 105, 250, 450, 550, 700, and 900ºC; with its mass being measured between each temperature interval. The percent mass lost at each interval in relation to the original mass of the sample represents the loss on ignition at that temperature. A Power-O-Matic 60 geotechnical oven (Blue M Electric

Company, Illinois, USA) was used for the 60 ºC and 105ºC intervals, with the crucibles being left for a period of at least 24 hours at each temperature. For the 250ºC and higher intervals high temperature furnaces (Lindberg/Blue M LGO Box Furnace and NCAT Asphalt Content Tester, Thermo Scientific, Massachusetts, USA) were used and crucibles were left for a minimum of 4 hours.

3.8. Specific Gravity Procedure

Due to the difference in density between organic and non-organic compounds, it was hypothesized that there may be some correlation between the BMP and the specific gravity of a waste sample. It was also hypothesized that the specific gravity data combined with LOI data might aid in identifying outliers for a BMP-LOI correlation. There are two ASTM standards for determining specific gravity of solid soil particles. ASTM D5550 is the simpler method, but ASTM D854 provides a more accurate measurement of specific gravity for substances that may contain soluble and/or floating particles.

MSW may contain both undesirable types of solids for specific gravity measurements by ASTM D854 (soluble and floating), and therefore ASTM D5550 is the preferred method for accurate determination of the specific gravity of waste. However, MSW is known to be far more variable than soil, and ASTM D5550 was deemed impractical and unnecessary for this research given the small increment in increased precision. Therefore, all specific gravity measurements were conducted based on ASTM D854. Measuring specific gravity by this method requires knowing the volume of water displaced by a known mass of solids. The most difficult and crucial part of the test is ensuring there is no air trapped between the solid particles at the time of measurement. The greater capillary action between fine particles make it more difficult to remove tiny bubbles. This problem is amplified by the fine, often fibrous components that are frequently found in MSW. Because of this, measuring the specific gravity of waste by this method requires greater care and more time than is usual for soils.

Pycnometers used for the specific gravity measurements were 500 mL volumetric flasks. Before each test, the mass of each flask filled with only de-aired water was measured and recorded. Each sample was tested in duplicate using between 25 and 110 g of shredded, dry material, or as much as was practical dependent on the nature of the waste. De-airing was achieved via a combination of heat and vacuum. High heat and vacuum were applied simultaneously for a short

period of approximately 5 minutes, until the water was just below boiling. After this point the heat was removed and the specimen remained under vacuum until further agitation produced no visible air bubbles, or for a period of at least 24 hours, at which point the volume was topped up to 500 mL with de-aired water and the mass was recorded. The specific gravity of the sample was then calculated according to the following equation:

$$G_s = \frac{M_s}{M_1 + M_s - M_2} \quad \text{(Eq. 3.2)}$$

Where: G_s = specific gravity; M_s = mass of the solid particles; M_1 = mass of the flask filled with de-aired water; and, M_2 = mass of the flask, solids, and water after de-airing.

3.9. BCP Equipment

3.9.1. Test Columns

Because the BCP test was designed to be aerobic in nature, it was imperative to maintain oxygen levels within the sample. This was achieved by selecting long and narrow columns as sample vessels, so that air bubbled up from the bottom of the sample would agitate and mix with the entire sample as it rose upwards, leaving no undisturbed portions in which anaerobic activity might initiate. Despite being an aerobic test, the columns were required to be sealed in order to properly monitor gas production/consumption rates. This meant that the air used to aerate the samples could not be introduced from the outside, but had to come from within the vessel itself. To achieve this, a single section of plastic tubing was attached from an opening in the headspace to an opening at the bottom of the column. A peristaltic pump was then used to constantly draw air from the headspace and force it down into the base of the sample. A small layer of coarse sand was placed at the base of the column to aid in dispersal of the air being bubbled through, and to prevent any solids from backing up into the aeration tubing. Another opening near the top of the column was fitted with two connectors for gas sampling and refreshing of the headspace using an automated respirometer. A fourth opening near the middle of the column was fitted with a connector that allowed any moisture extracted during gas sampling to be reintroduced. Figure 3.7 shows a specially constructed base with four of the BCP columns mounted to it.

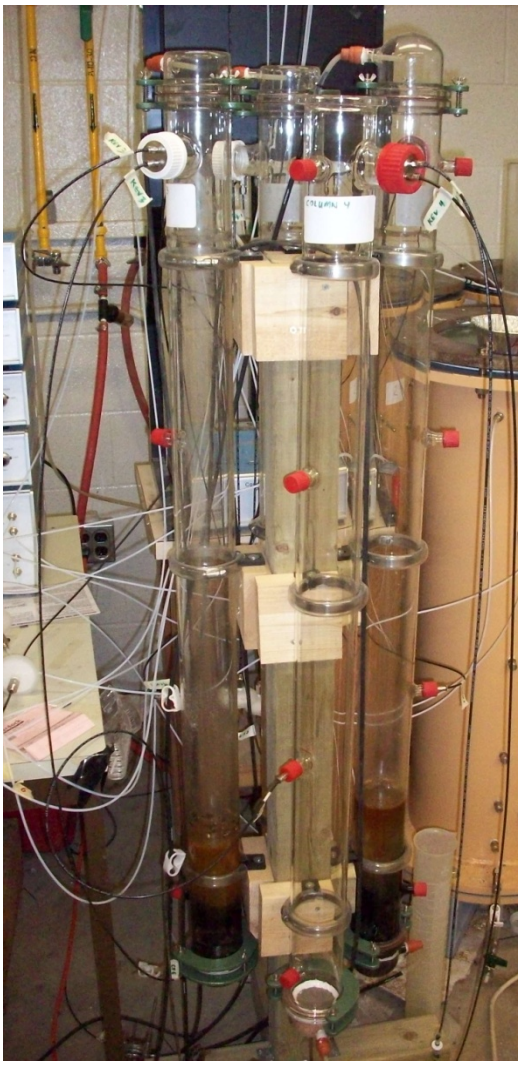


Figure 3.7: BCP test setup showing four BCP columns mounted to a single base, with tubing connections running between each column and the Micro-Oxymax.

3.9.2. Micro-Oxymax

An automated respirometer (Micro-Oxymax, Columbus Instruments, Ohio, USA) was used to continuously monitor both CO_2 production and O_2 consumption, throughout the duration of the test. The Micro-Oxymax is a lab instrument designed for automated monitoring of respiration activity, making it ideal for use in the BCP test. Its modular design allows monitoring of up to three gases by linking individual modules in series. Many modules are available and each is capable of measuring one type of gas within a specific range of concentration. Other modules increase the number of channels available, allowing simultaneous monitoring of many individual vessels. The gas modules used for this research were a 0-100% O_2 sensor, a 0-10% CO_2 sensor,

and a 0-10% CH_4 sensor. Because the Micro-Oxymax requires dry gas for optimal operation, all sample gases are fed through a condenser, followed by multiple moisture traps before reaching the sensors. Any water extracted from the gas by the condenser is gravity fed back into the sample vessels.

The great advantage of the Micro-Oxymax is its automation. Since it not only measures gas concentrations, but also volumes, once it is running the test requires no further interference by the user. The machine will even periodically refresh the headspace in the vessels; ensuring aerobic conditions are maintained. It does this whenever user-defined concentration thresholds are detected.

3.10. BCP Procedure

The Micro-Oxymax was preprogrammed, and properly hooked up to each test column prior to starting the tests. Each BCP test used 100 g of shredded MSW material, 1000 mL of distilled water, and 10 mL of prepared BOD seed as inoculant. These components were placed into the columns and mixed thoroughly using a long rod. The columns were then sealed and the peristaltic pump turned on to begin aeration. The last step was activating the Micro-Oxymax. Once activated, the Micro-Oxymax automatically took gas composition and volume readings every 6 hours. The headspace of each column was refreshed automatically whenever CO_2 concentrations within that column exceeded 4%. The Micro-Oxymax software calculated individual gas concentrations and volumes at every time interval, as well as tracking cumulative gas production/consumption over the course of the entire test. All of this information could be accessed on the fly at any point during testing.

3.11. Summary

This study used materials from four separate sources, each with its own unique characteristics. Each material was processed according to its individual requirements. The three tests performed on all materials were BMP, sequential LOI, and Specific Gravity. A first attempt at developing a BCP test was attempted on a select few samples. The equipment and procedures required for all tests were outlined.

Chapter 4 Presentation of Results

4.1. Introduction

This chapter presents the results of the testing program. Data is presented for each of the tests outlined in Chapter 3. This includes ultimate BMP values, sequential LOI values at each temperature, and the determination of the specific gravity for each test sample. Results of the initial attempt at the aerobic BCP test are also given.

4.2. Results of BMP Testing

4.2.1. Individual Test Set up Details

The exact details of the BMP setup were not identical between test batches. The setup details for each sample are given in Table 4.1. Exact sample masses were not always precisely equal between replicate samples, sometimes varying by a few tenths of a gram. Calculation of BMP values for each replicate sample were done using the precise measured mass (corrected for residual water content) of that sample. The gravimetric water contents shown are approximate because the water content of the inoculum was never tested. For the purposes of calculating the gravimetrc water content of the test samples, the inoculum was assumed to be 90% water by volume, and the solids were assumed to have a specific gravity of 2.0.

Table 4.1: Details of modified BMP setup for each sample. Testing for each sample listed consisted of at least three replicate sets, each possessing the same setup details.

	Sample	Mass of Sample (g)	Volume of Water Added (mL)	Volume of Inoculant Added (mL)	Resulting Headspace Volume (mL)	Approximate Gravimetric Water Content
Barrie	M13	200	100	100	2040	0.86
	M14	200	100	100	2040	0.86
	M15	200	100	100	2040	0.86
	M17	200	100	100	2040	0.86
RMWB	BH1A	200	900	200	1130	4.5
	BH2A	200	1200	200	830	5.8
	BH2B	200	1200	200	830	5.8
	BH3A	200	900	200	1130	4.5
	BH4A	200	900	200	1130	4.5
	BH5A	200	900	200	1130	4.5
	BH6A	200	900	200	1130	4.5
	BH7A	200	1200	200	830	5.8
	BH8A	200	900	200	1130	4.5
	BH9B	200	1100	200	930	5.3
	BH10A	200	900	200	1130	4.5
	BH11A	200	1100	200	930	5.3
	BH12A	200	1100	200	930	5.3
	BH13A	200	1100	200	930	5.3
	BH14A	200	1100	200	930	5.3
	BH15A	200	1100	200	930	5.3
	BH16A	200	1100	200	930	5.3
	BH17A	200	1100	200	930	5.3
	BH18A	200	1100	200	930	5.3
	BH18B	200	1100	200	930	5.3
	BH19A	200	1100	200	930	5.3
	BH20A	200	1100	200	930	5.3
Spadina	S1	200	1500	150	580	7.1
	S2	200	1000	150	1080	4.9
	S3	100	1000	200	1080	8.4
	S4	100	1000	150	1130	8.7
	S5	200	1000	150	1080	4.9
	S6	200	1000	150	1080	4.9
	S7	200	1000	150	1080	4.9
	S8	200	1000	150	1080	4.9
	S9	50	1500	150	655	20
	S10	200	1000	150	1080	4.9
Cowlitz	D	200	1000	200	1030	4.9
	W	200	1000	200	1030	4.9
	A	200	1000	200	1030	4.9
	CS	200	1000	200	1030	4.9
	CS/MSW	200	1000	200	1030	4.9

The Barrie samples were the first tested, and served as a trial run of the modified BMP method. In the first attempt most vessels developed several large leaks at similar locations on the test vessels, so the results were thrown out and a second attempt was made. This time the problem areas were preemptively sealed with silicone. The results of the second attempt were acceptable, though in hindsight, the tests were perhaps terminated a bit prematurely. The mixtures were also noticed to be quite viscous and difficult to agitate by shaking alone. For this reason the volume of water in all following tests was significantly increased. Since gas was only removed from the sample bag, headspace volumes were required to calculate the amount of gas remaining in the vessel between each reading. The total volume of the vessels (without gas sample bag) was measured as 2330 mL. The volume of the waste material was calculated based on an average specific gravity of 2.0, which was first estimated using simple principles of water displacement in only a few trials, and later confirmed through rigorous specific gravity testing of all samples (results presented in Section 4.4).

4.2.2. Ultimate Methane potentials

The final BMP results of all samples are given in Table 4.2. BMP values presented are based on the ultimate BMP values determined from the fitted curves described in Chapter 5. The coefficient of variation (CV) is the standard deviation divided by the mean. It was calculated for each sample from all successfully completed replicate tests within that sample. A lower CV value indicates greater agreement between replicate samples.

The results all fall well within the range of BMP values reported for MSW in the literature. It is clear when viewing these results alongside Table 4.1, that the nature of the material (composition, age, etc.) has a much greater effect on the final results than the exact details of the setup. Several plots of methane potential over time are given later in this chapter. Similar plots for all samples may be found in Appendix B. Inspecting these plots reveals that variables such as gas production rate, and time to completion, also appear to have a much greater dependence on the material, rather than the other factors tested. The large CV values of samples S7 and A are due to the measured BMP values being very low. In cases like those, an increase of only one or two mL/g can result in values that are different by a factor of two or three, which results in a very large CV, despite very similar results. This suggests that a more precise method may be better suited to materials expected to possess very low BMP.

In most cases, the amount of oxygen remaining in the vessels at the beginning of the test was negligible, and none was detected. In a few cases, residual oxygen was detected in the vessels during the first one or two measurements. In these cases the concentration of CO_2 was measured to be greater than the concentration of methane until the residual oxygen was depleted. An evaluation of the impact of this residual oxygen on the ultimate BMP values was conducted by adjusting the measured CO_2 and methane concentrations to be equal (this adjustment has not been included in the reported ultimate BMP values). In the worst case, it was found that the residual oxygen in the test vessels may have caused the ultimate BMP to be underestimated by as much as 1.4 mL/g, though in most cases it was much less.

Table 4.2: Maximum, minimum, and average ultimpate BMP results from successful replicates of each test.

Sample	Number of Successful Replicate Tests	Maximum BMP_{ult} (L/kg)	Minimum BMP_{ult} (L/kg)	Average BMP_{ult} (L/kg)	CV (%)
M13	3	1.91	1.29	1.80	20.9
M14	3	12.7	6.83	9.30	32.3
M15	3	6.76	6.17	6.47	6.47
M17	3	8.94	6.11	7.44	19.1
BH1A	3	34.3	30.8	32.2	5.75
BH2A	3	31.5	22.7	26.3	17.4
BH2B	4	32.7	20.0	27.0	20.1
BH3A	3	11.6	9.61	10.5	9.69
BH4A	3	7.37	6.28	6.71	8.60
BH5A	3	13.4	11.5	12.7	8.52
BH6A	3	26.6	25.1	26.1	3.28
BH7A	3	46.1	45.2	45.7	1.11
BH8A	3	24.7	17.9	22.3	17.1
BH9B	2	43.8	38.3	41.0	9.58
BH10A	5	32.6	26.6	29.2	7.46
BH11A	3	26.9	23.2	24.7	7.91
BH12A	3	14.0	12.9	13.3	4.61
BH13A	3	41.0	36.7	38.8	7.83
BH14A	3	5.82	5.20	5.56	5.76
BH15A	3	17.9	16.0	17.0	5.74
BH16A	3	41.1	35.7	38.1	7.18
BH17A	3	17.4	12.0	15.4	19.1
BH18A	3	6.00	3.93	5.23	21.7
BH18B	3	3.36	2.28	2.83	19.1
BH19A	3	22.4	14.2	19.0	22.7
BH20A	3	1.75	1.20	1.45	19.2
S1	3	72.0	55.0	64.1	17.4
S2	3	7.62	6.08	6.94	11.3
S3	3	139	138	138	0.410
S4	3	93.7	84.5	89.1	7.25
S5	3	3.75	2.83	3.41	14.7
S6	3	4.69	3.28	4.07	17.8
S7	3	4.88	1.30	2.76	67.9
S8	3	10.8	8.62	9.62	11.7
S9	3	5.83	5.30	5.53	4.93
S10	3	20.3	16.3	18.3	15.4
D	3	71.9	50.5	63.5	17.9
W	3	79.8	57.0	68.4	23.6
A	3	4.60	2.67	3.63	37.7
CS	3	61.1	59.0	60.1	2.48
CS/MSW	3	55.4	42.7	49.2	13.0

4.2.3. Inhibition Effects

Early in the testing program, it was noticed that certain vessels would experience a sudden drop in gas production rate. Often these vessels would simply stop producing gas entirely, but occassionally production would pick up again after a few days. The sources and mechanisms of inhibition within anaerobic digestion processes are considerably varied and complex (Chen et al. 2008), making them very difficult to predict. Testing for and identifying the precise causes of inhibition in the affected vessels would not have contributed a great deal to this research, plus the time and energy required would have been prohibitive; thus the reasons for these events remain unknown. With the exception of sample BH9 (which experienced inhibition in 4 out of 6 attempts), inhibition showed no correlation to either sample material or inoculum. One of the reasons for using three replicate samples rather than two was in case one of the vessels experienced sudden inhibition and ceased producing gas. In these cases the results from the offending vessel were not included in the final analysis. If inhibition was detected early during the test, the offending vessel could simply be dismantled and replaced, but given the lengthy time requirement for BMP testing, this was not always a practical option if inhibition occurred at a later point.

4.2.4. Reliability Checks

Several tests were performed to ensure that there were no systematic errors in either the sample processing or testing that might produce erroneous results. As described in Chapter 3, each borehole core from RMWB was divided into two replicate subsamples (A and B). To determine the validity of this division, all tests performed on sample BH18 were conducted on both subsamples, individually. Figure 4.1 shows the results for BMP testing on both subsamples of BH18. Results for the sequential LOI and specific gravity tests can be found later in this chapter; in Table 4.5 and Table 4.7 respectively. A Student's t-test (two-tailed, assuming equal variance) performed on the test results of the two subsamples indicates that there is a statistically significant difference ($p < 0.05$) between the subsamples in all tests. However, this does not indicate the magnitude of the difference, only that there was a consistent difference. The mean values of BMP for the two subsamples differ by only 2.4 L/kg, which is much less than the difference between the maximum and minimum values measured in replicate tests of many other samples. Overall, the effect of the subsampling was not considered to have a large outcome on the final result of the test. It should be noted however, that this outcome (produced using sample masses of 200 g) does

provide further evidence of the large variability of MSW, and supports the hypothesis that sample masses on the order of 1 g to 10 g are likely not to be representative.

Because inoculum was gathered as needed from the local wastewater treatment plant, there was concern minor temporal variations in the microbial cultures at the time of collection of inoculum samples may have an effect on the results. This was tested by performing the BMP test on sample BH10A in two triplicate sets, using two different batches of inoculum, separated by nearly a year. The results are shown in Figure 4.2. Unfortunately, the third replicate of the first set (BH10A-C) experienced random chemical inhibition and was discontinued. The results of a t-test comparing the five reliable data points indicate that there is a 49.9% chance that the means of the two data sets are equal. Therefore we can safely conclude that minor variations in the inoculum, due to batches being collected at different times, have no appreciable effect on the outcome of the results.

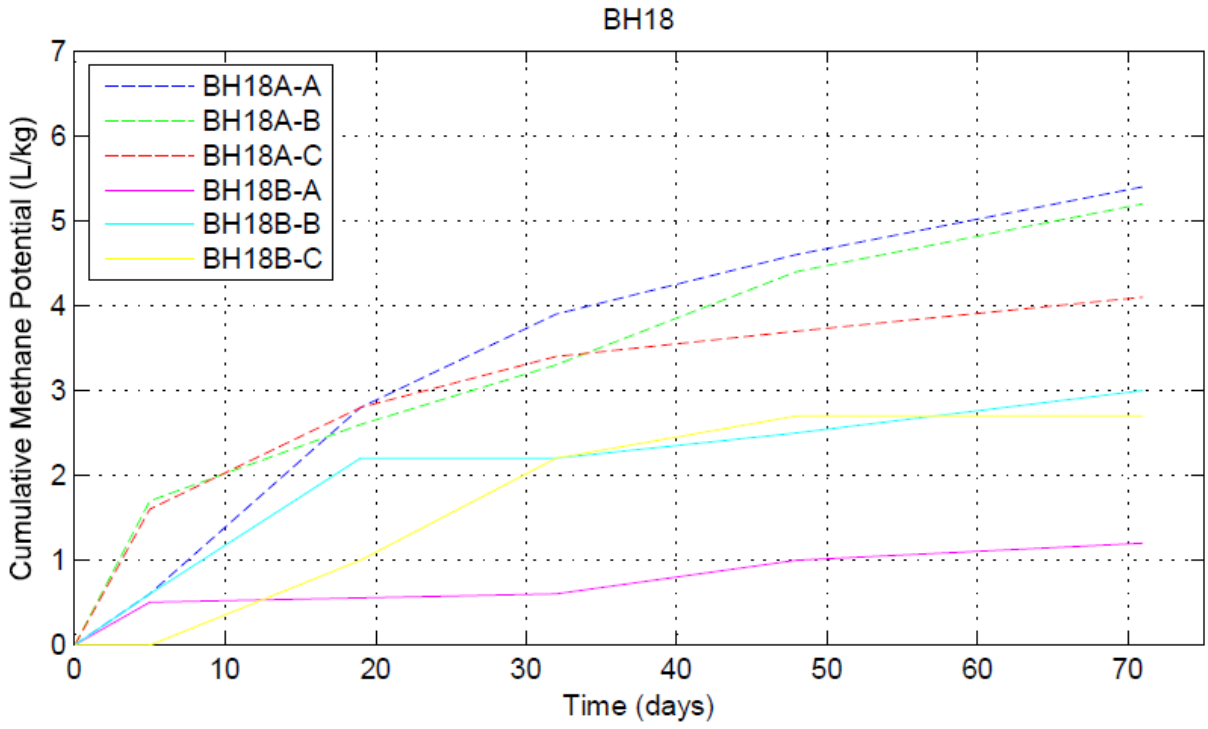


Figure 4.1: Comparing cumulative methane potential curves for samples BH18A and BH18B; the two subsamples of sample BH18.

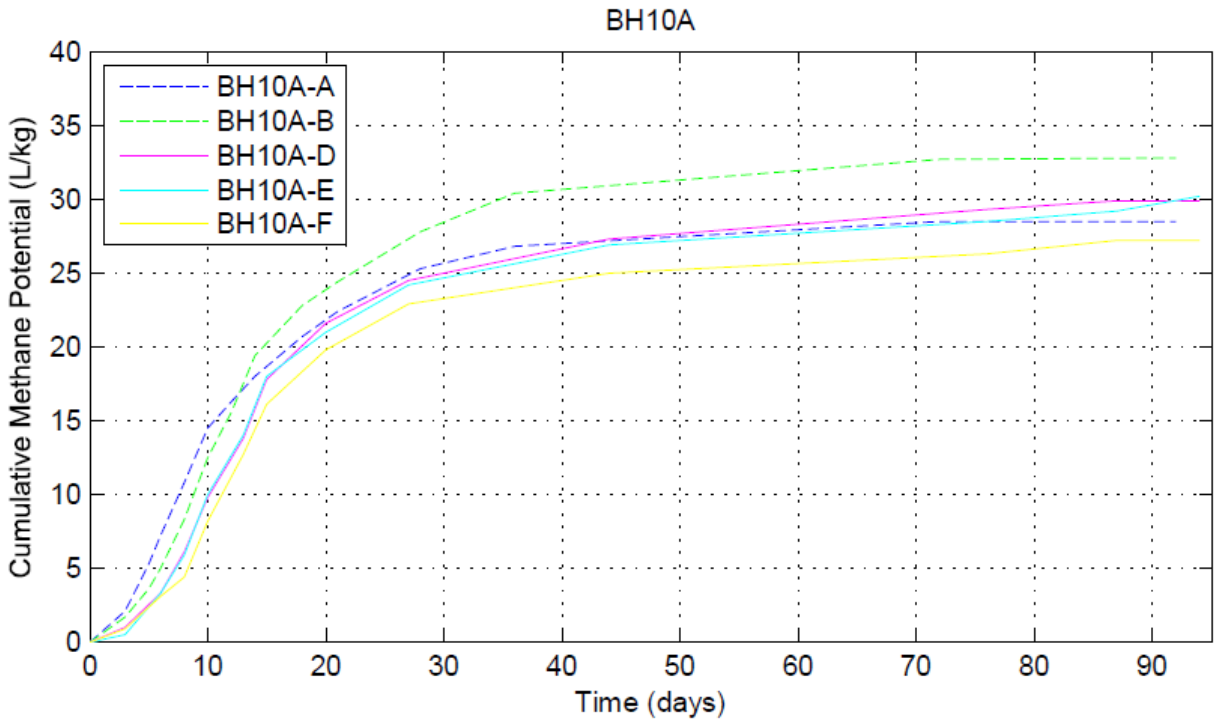


Figure 4.2: Comparing cumulative methane potential curves of BH10A replicate tests. BH10A-D, BH10A-E, and BH10A-F were tested one year following BH10A-A and BH10A-B, using a separate, freshly collected batch of inoculum.

In the case of sample BH2, a combination of the above two scenarios was tested. Because there was not enough of subsample BH2A to perform all tests, BH2B was later tested using a separate batch of inoculum. The results are shown in Figure 4.3. This time, something about the inoculum or the material caused a change in the rate of gas production. Samples from BH2A exhibited dual phase gas production behaviour, while samples from BH2B did not. The ultimate BMP values determined however, showed no statistically significant difference between the two sets of tests. A t-test performed on the data showed that there is an 81.5% chance that the means of the two data sets are the same. This strengthens the conclusion that the quartering and subsampling procedure does not have an appreciable effect on the ultimate BMP values measured, despite the statistically significant difference detected between BH18A and BH18B.

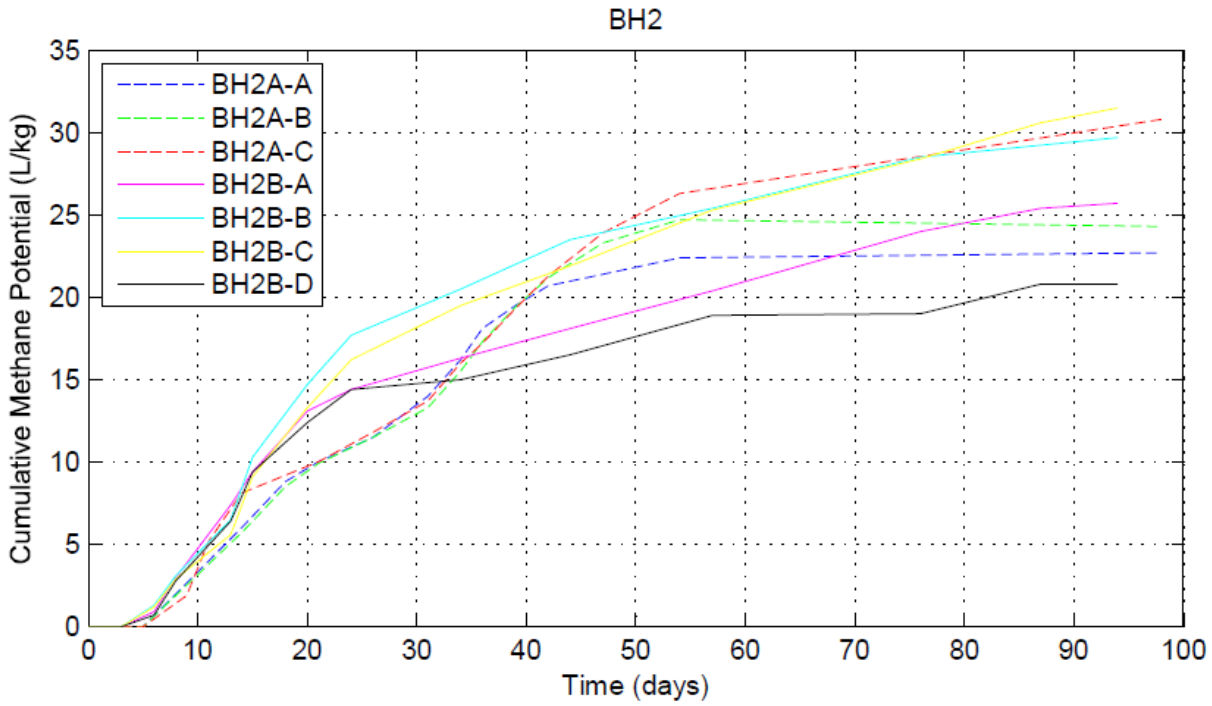


Figure 4.3: Comparing cumulative methane potential curves of BH2A and BH2B. These two subsamples of BH2 were tested individually, using two separate batches of inoculum.

An internal check on the accuracy of the modified BMP method was also performed. As explained in Chapter 2, the last step in the anaerobic degradation process is the conversion of acetate into CO_2 and CH_4. The reaction may be described as:

$$CH_3COO^- + H^+ \rightarrow CH_4 + CO_2 \qquad \text{(Eq. 4.1)}$$

From this relatively simple equation, it is possible to theoretically calculate the exact volume of gas that should result from introducing a known amount of acetate into a BMP vessel. This was done to the triplicate set of sample BH7A after gas production had slowed enough to be considered the ultimate BMP. Precisely 21 mL of a 1 molar sodium acetate solution was added to each vessel, and it was calculated that this should produce exactly 1010 mL of gas. The amount of gas recovered is presented in the frist part of Table 4.3. It is possible to double check this by tracking the carbon that is added to the system, since all of the carbon added should be released in the form of CH_4 and CO_2. The results of that check are provided in the second part of Table 4.3.

Table 4.3: Results of internal accuracy check of BMP testing by adding 21 mL of 1 M sodium acetate solution to BH7A. Part a) tracks the volume of gas produced compared to what was expected based on stoichiometry and the ideal gas law. Part b) tracks the moles of carbon that were recovered compared to what was introduced based on gas composition measurements.

a) By Volume of Gas

Sample	Theoretical Volume of Gas Produced (mL)	Measured Volume of Gas Produced (mL)	% Recovery
BH7A	1000	900	90%
BH7B	1000	811	81%
BH7C	1000	1014	101%

b) By Moles of Carbon

Sample	Moles of C Added	Total moles of C Produced	% Recovery
BH7A	0.042	0.038	90%
BH7B	0.042	0.034	82%
BH7C	0.042	0.042	101%

Theoretically, the two calculations should show exactly the same amount of recovery. The error between them can likely be attributed to the temperature assumption required when determing the recovery by moles of carbon. Some of the error in total recovery can be explained by the precision of measurements, both in preparing the sodium acetate solution and in injecting it into the vessels. Overall the results are within acceptable margins of error. They indicate that the processes taking place within the BMP vessels are as they should be, and serve to help validate this BMP method.

Lastly, an external check was completed by sending 15 samples to a lab at North Carolina State University (NCSU). The points below present some of the key differences between the NCSU BMP method, and the method used in this study:

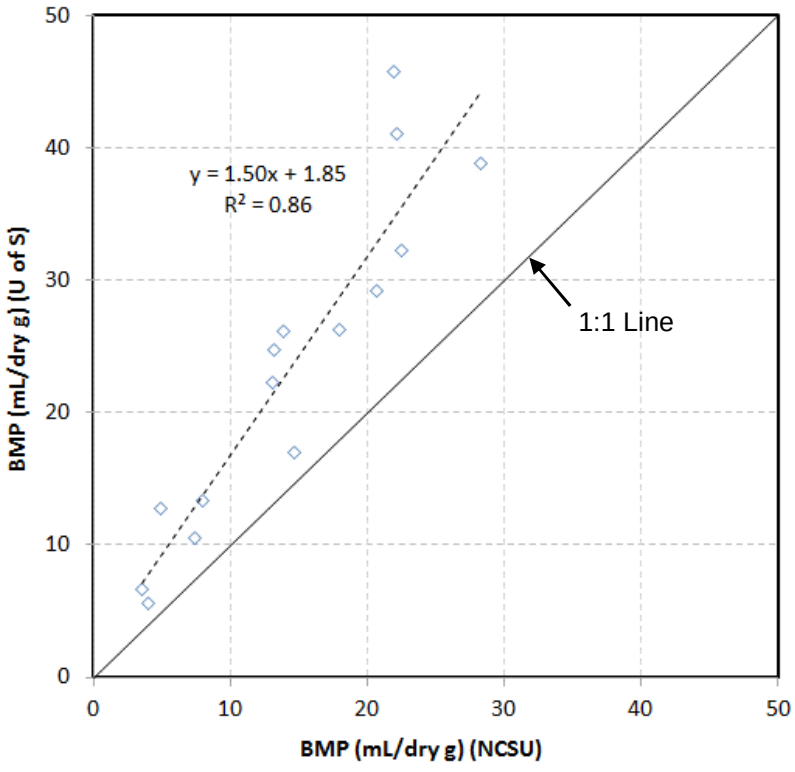
- Only a few grams of sample are used;
- test duration is limited to 60 days;
- a nutrient medium is added to the vessels to promote microbial activity;
- test vessels are allowed to overpressurize, and gas is collected via a syringe through a rubber septum; and,
- gas is analyzed using a GC.

A comparison of the results from this study (U of S), and those from NCSU are given in Table 4.4 and plotted in Figure 4.4.

Table 4.4: Comparison of BMP_{ult} results between the modified method presented in this study (U of S), and the method used by NCSU.

Sample	BMP_{ult} (mL/dry g) (U of S)	BMP_{ult} (mL/dry g) (NCSU)	CV (U of S)	CV (NCSU)
BH1A	32.2	22.5	5.75	7.92
BH2A	26.3	18.0	17.4	7.39
BH3A	10.5	7.42	9.69	27.2
BH4A	6.71	3.51	8.60	9.60
BH5A	12.7	4.90	8.52	9.89
BH6A	26.1	13.9	3.28	117
BH7A	45.7	22.0	1.11	8.53
BH8A	22.3	13.1	17.1	8.99
BH9B	41.0	22.2	9.58	11.4
BH10A	29.2	20.7	7.46	16.5
BH11A	24.7	13.2	7.91	8.55
BH12A	13.3	8.00	4.61	5.24
BH13A	38.8	28.3	7.83	1.71
BH14A	5.56	3.96	5.76	16.4
BH15A	17.0	14.7	5.74	8.02
		Average CVs	8.02	10.6

Figure 4.4: Plot comparing the BMP_{ult} values of the modified method presented in this study (U of S) to those of the method used by NCSU.



The straight black line is a 1:1 line added for reference purposes. The fact that all of the values from this study average 1.50 times greater than the values from NCSU is most likely due to the unlimited incubation time allowed by the U of S method. Another possibility is bias in the measurements made by the GEM, causing readings to be consistently higher than reality. Bias is unlikely however because of the regular calibrations using standard gas mixtures. The concentrations of the standard gas mixtures used were also chosen to be very near the concentrations being measured, further increasing the confidence that instrument bias can be ruled out.

Since instrument bias has been ruled out, the consistently higher BMP values, and fairly linear distribution both serve to indicate that the BMP method used in this study is not only valid, but may be superior in terms of accuracy to that used by NCSU. The slightly lower CV value also suggests that the two tests are similar in terms of precision, supporting the hypothesis that taking multiple readings from a less precise instrument, and averaging the results, can produce equal or greater precision to a single reading from a more precise instrument. A t-test applied to the two sets of CV data predicts that there is a 51.2% chance of the average CV values being the same.

4.3. Results of Sequential LOI Testing

Results of the Sequential LOI tests are given in Table 4.5. Each sample was tested in triplicate at each temperature, but in the interests of clarity and conciseness, only average values are reported here. The large majority of CV values for triplicate sets were less than 5%, and only a very few were above 15%. Figure 4.5 provides a graphical representation of LOI results for a typical sample. Similar figures for all samples can be found in Appendix D.

The values vary widely between the different samples, but all are within expected ranges, based on age and composition. For every sample tested, the largest jump in mass loss is between 105ºC and 250ºC, with a second smaller jump between 250ºC and 450ºC. In most cases the increase in mass loss at temperatures above 450ºC is minor compared to these two jumps. The two samples showing the greatest increase in mass loss between 250ºC and 450ºC are S9, and A, the two samples with the highest plastic content. This corresponds to the auto-ignition temperature of several common plastics and other non-biodegradable organic polymers, as shown in **Error! Reference source not found.**. This fact may be useful in determining a correlation between BMP and LOI.

Table 4.5: Summary of results of sequential LOI testing for each sample.

Sample	Average LOI @ 105ºC (%)	Average LOI @ 250ºC (%)	Average LOI @ 450ºC (%)	Average LOI @ 550ºC (%)	Average LOI @ 700ºC (%)	Average LOI @ 900ºC (%)
M13	0.22	4.43	7.83	8.18	12.61	15.99
M14	0.30	6.12	11.76	12.21	16.44	19.80
M15	0.25	5.05	9.21	9.80	13.60	18.87
M17	0.33	7.54	11.92	12.38	16.47	20.48
BH1A	0.54	28.29	33.52	33.84	35.27	35.39
BH2B	1.04	32.05	37.08	37.68	39.29	39.67
BH3A	0.61	19.00	25.19	26.21	28.73	28.41
BH4A	0.36	12.24	18.21	18.78	20.64	20.72
BH5A	0.40	17.55	23.45	23.93	26.04	26.17
BH6A	0.84	25.01	29.67	29.97	31.77	31.83
BH7A	1.32	46.97	52.88	53.08	54.40	54.16
BH8A	1.38	31.36	35.71	36.44	38.39	38.69
BH9B	1.29	37.65	41.89	42.32	43.70	43.98
BH10A	0.80	17.02	24.79	25.65	27.19	27.49
BH11A	0.64	22.88	28.82	29.10	30.25	30.46
BH12A	0.41	12.87	18.00	19.63	21.43	21.66
BH13A	0.85	35.94	40.64	41.59	42.70	43.15
BH14A	0.46	15.07	20.17	20.92	22.97	23.03
BH15A	0.40	16.23	21.51	22.90	26.61	26.61
BH16A	0.92	25.10	30.73	31.62	32.90	33.06
BH17A	0.61	16.21	19.44	20.57	24.17	24.96
BH18A	0.63	11.66	16.35	17.48	19.97	20.01
BH18B	0.40	10.16	12.69	13.56	16.13	16.28
BH19A	0.52	17.30	19.72	20.62	23.43	23.64
BH20A	0.25	3.46	6.20	6.70	10.09	10.85
S1	0.81	68.90	74.94	75.44	76.27	76.43
S2	0.34	13.97	18.91	20.89	24.72	25.01
S3	0.96	81.23	84.84	85.17	85.69	85.79
S4	0.72	42.27	47.96	49.10	51.23	51.55
S5	0.57	6.99	12.89	13.55	17.02	18.67
S6	0.57	7.88	14.56	15.45	18.73	19.82
S7	0.57	7.95	14.85	15.75	19.21	20.61
S8	0.58	9.04	15.97	16.67	20.40	22.14
S9	0.37	11.64	76.80	77.55	77.96	78.24
S10	0.59	48.53	55.06	55.67	58.37	58.79
D	0.71	46.80	52.83	53.70	63.08	66.79
W	1.62	48.16	55.02	55.66	65.57	68.65
A	0.45	34.38	52.95	53.22	54.3	54.58
CS	0.94	40.41	49.78	50.63	58.1	58.28
CS/MSW	0.88	29.82	38.1	39.01	43.58	43.84

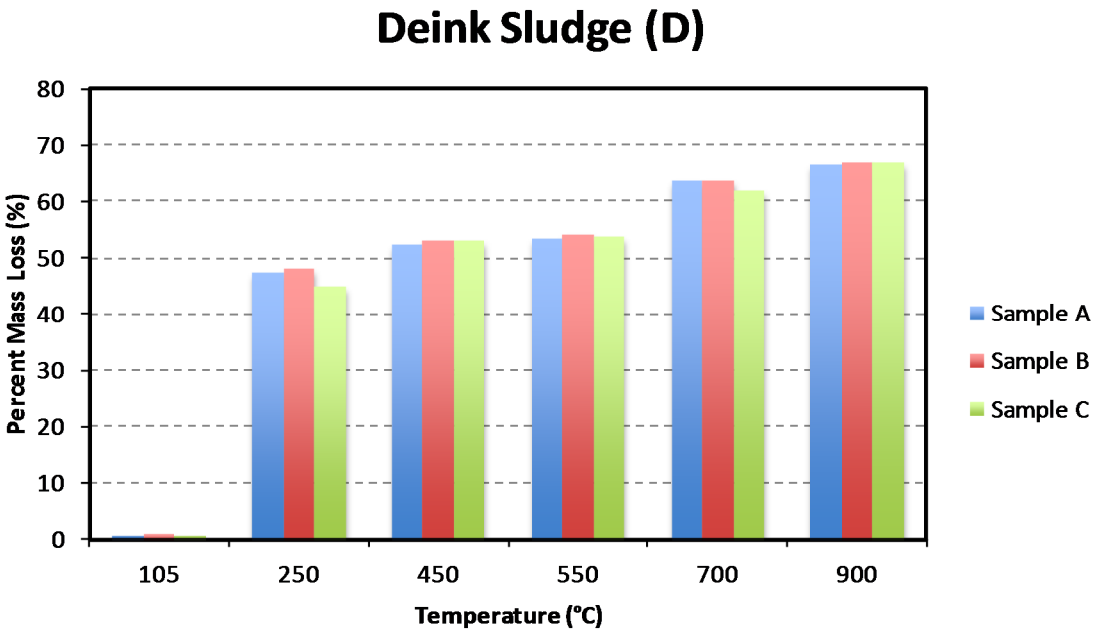


Figure 4.5: Typical graphical representation of sequential LOI results.

Table 4.6: Ignition temperatures of common plastics and other non-biodegradable organic polymers (Cafe, 2007).

Material	Auto-Ignition Temperature (ºC)
LDPE/HDPE	349
ABS	416
PVC	435-557
Polyesters	432-488
Polyurethanes	416
Nylons	424-532
Rubber	260-316

4.4. Results of Specific Gravity Testing

Specific gravity testing was initiated after the samples from Barrie had already been disposed of. For the remaining samples, specific gravity tests were conducted in duplicate, and the results are shown in Table 4.7.

Table 4.7: Summary of results of specific gravity testing.

Sample	Average G_s	Coefficient of Variation (%)	Sample	Average G_s	Coefficient of Variation (%)
BH1A	2.01	2.50	BH18B	2.31	0.09
BH2B	1.92	1.15	BH19A	2.27	1.43
BH3A	2.19	0.04	BH20A	2.49	1.85
BH4A	2.30	0.06	S1	1.57	1.71
BH5A	2.18	1.60	S2	2.30	1.87
BH6A	2.10	0.34	S3	1.62	3.28
BH7A	1.72	3.77	S4	1.84	5.56
BH8A	2.01	0.32	S5	2.37	0.33
BH9B	1.92	2.75	S6	2.33	0.31
BH10A	2.01	1.30	S7	2.31	0.25
BH11A	2.12	2.32	S8	2.16	2.46
BH12A	2.21	0.46	S9	1.19	10.23
BH13A	1.98	1.51	S10	1.75	0.88
BH14A	2.29	0.66	D	1.83	0.88
BH15A	2.25	2.32	W	1.95	6.90
BH16A	2.07	2.61	A	1.57	1.50
BH17A	2.24	1.60	CS	1.71	1.92
BH18A	2.24	1.33	CS/MSW	1.94	0.01

For all tests, agreement between the replicate samples was very good. This adds confidence to the assumption that ASTM D854 is an appropriate method for testing specific gravity of MSW. The average specific gravity across all samples was 2.04, with a coefficient of variation of 13.9%.

4.5. Results of the BCP Trial

Three samples were chosen to test the efficacy of the proposed BCP method. The samples were chosen based on their BMP results: BH7A was expected to have a high potential; BH20A was expected to have a low potential; and, BH11A was expected to have a mid-range potential. Also, none of these samples suffered from unexpected inhibition, and all had reasonable agreement between their replicates during BMP testing. The results of the trial are plotted in Figure 4.6.

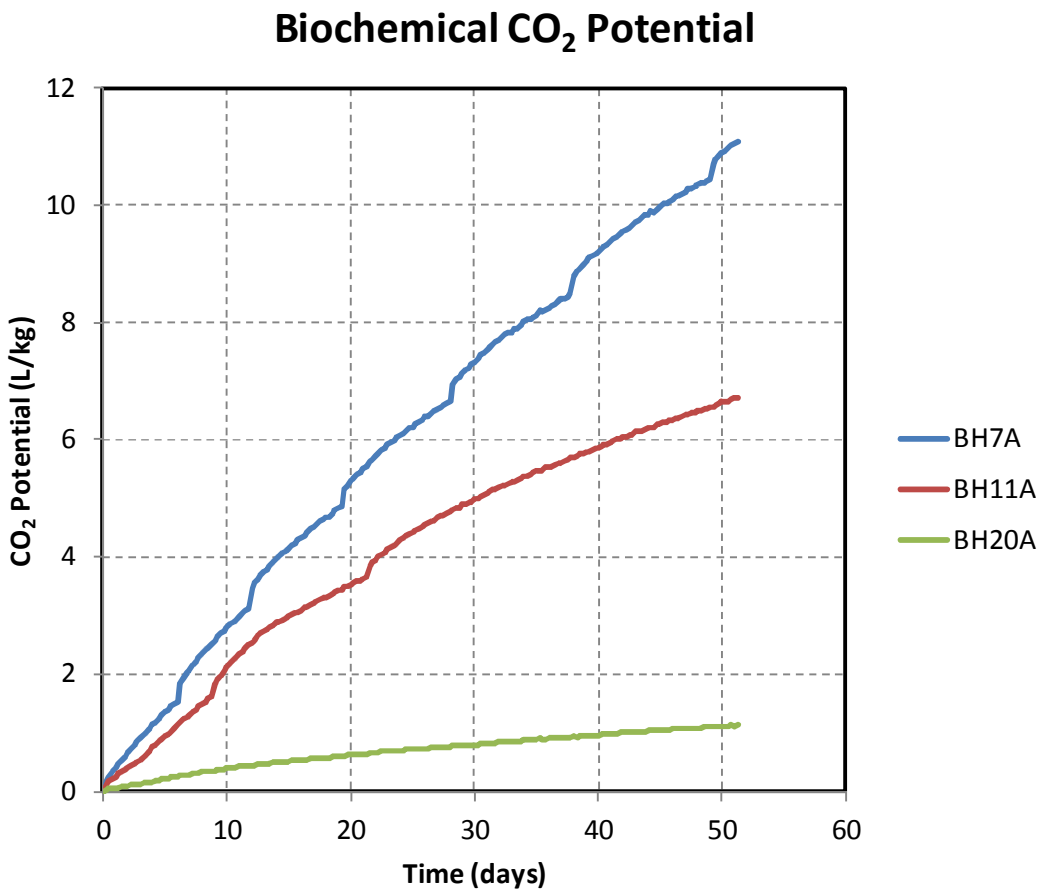


Figure 4.6: CO_2 potential vs. time for the three samples tested in the BCP trial.

The jumps in the curve correspond to the automatic refreshment of the headspace once a threshold value of CO_2 was detected by the Micro-Oxymax. Overall, the test appears to have worked. The CO_2 potentials appear to mirror the methane potentials in that the low methane potential sample has a low CO_2 potential, and likewise for the mid-range and high BMP potential samples. The main goal of developing the BCP method however, was to significantly reduce the time required for testing. Expected time requirement was in the vicinity of 30 days. This trial was allowed to run for 52 days, at which time it still showed no signs of reaching an ultimate potential. Given the added complexity of this test relative to the alternatives (such as LOI), no further trials of the BCP approach were made.

The trial was by no means perfect, and there is definite room for improvement. A more efficient air circulation system, the adjustment of the relative concentration of nutrients, adjusting

the volume of water, etc., may all have positive effects on any future trials which may be undertaken.

4.6. Summary

The results of the tests described in Chapter 3 were presented in this chapter. Details of the BMP experiments were given, and the validity of the method used was evaluated. The sequential LOI tests revealed that mass loss at temperatures below 550ºC might be of some use for waste characterization. Also, ASTM D854 was shown to be a consistent method of specific gravity determination for MSW. Finally, the first attempt at performing a BCP test was shown to be mostly unsuccessful, but with some promise for future trials.

Chapter 5 Analysis of Results

5.1. Introduction

This chapter presents a detailed analysis of the results presented in Chapter 4. An investigation was made into methods for predicting ultimate BMP values from the data collected regarding the cumulative volume of CH_4 over time. The ultimate BMP values reported in the previous chapter will also be compared to various other values. Relationships are shown to exist between ultimate BMP, LOI, and specific gravity.

5.2. First-Order Decay Model

It has been found that methane production during a BMP test tends to follow a first-order decay rate (Gunaseelan, 2004; Gregory & Browell, 2011; Raposo et al. 2011). One simple form of the first-order decay equation for modelling methane potential is as follows:

$$\boldsymbol{BMP(t) = BMP_{ult}\left(1 - e^{-k(t-t_{lag})}\right)} \qquad \text{(Equation 5.1)}$$

Where: BMP(t) = the BMP at time, t; BMP_{ult} = the ultimate BMP; k = the decay constant; and, t_{lag} = the lag time at the beginning of the test before microbial activity reached its peak.

The numerical computation program MATLAB® (MathWorks Inc., Natick MA, USA) was used to produce the fitted curves. Plots of all the fitted data can be found in Appendix B. A table of all calculated curve fitting parameters (BMP_{ult}, k, and t_{lag}) can be found in Appendix C.

It should be noted that some of the t_{lag} values reported are negative. Mathematically, this would indicate that the sample digestion was already underway at the time that test monitoring began. That is to say that a methane potential greater than zero was recorded at time zero. Clearly, the data shows that this was not the case, and these results are merely a mathematical product of the curve fitting procedure. An investigation of the average k values calculated from the curve fitting procedures also revealed no relationship to either BMP_{ult} or type of material. No example

could be found of either of these parameters being reported in literature, and they have been included here only for completeness, and to aid others who may wish to verify and/or replicate this research.

5.2.1. Determining the End of the Lag Phase

The standard first order decay model does not account for the lag time at the beginning of the test. Therefore, in order to achieve the best fit possible, several data points at the beginning of each test were not included in the curve fitting procedure. The following procedure was developed to objectively determine how many points from the beginning of the test should be excluded from the fitted curve:

1. Since the methane production curve should ideally show an ever-decreasing gas production rate (slope of the curve), compute the production rate between every two consecutive points in the data set.
2. Starting with t = 0 and working forward chronologically, identify the point at which the gas production rate first shows a decrease.
3. The point immediately preceding the point identified in Step 2 is noted as the *alpha* point.
4. Compute the percent change in gas production rate between the *alpha* point and every subsequent point.
5. If a subsequent point possesses a gas production rate more than 80% greater than the rate at the *alpha* point, that point is noted as the new *alpha* point.
6. Repeat Steps 4 and 5 as many times as necessary until a final *alpha* point is identified. This should be the first point included in the fitted curve. All preceding points should be omitted from the fitted curve.

Steps 4, 5, and 6 are required because noise in the data sometimes causes gas production rates to fluctuate between increasing and decreasing. The threshold value of 80% was chosen after a process of trial and error revealed it to produce the most satisfactory results. The implementation of this procedure is perhaps more clearly explained using an example. Table 5.1 provides an example of the procedure using data from BH7A-A.

Table 5.1: Example showing the methodology for the determination of the end of the lag phase, using results from sample BH7A-A.

t (days)	BMP (L/kg)	Gas Production Rate (L/kg/day) [Step 1]	% Change in rate from t = 3 [Step 4]	Notes
0	0.00	0.257	-	
3	0.770	0.765	-	α_1 [Step 3], Superseded by α_2 [Step 5]
5	2.30	0.611	-20.1	First decrease in gas production Rate [Step 2]
6	2.91	0.732	-4.27	
8	4.38	0.900	17.6	
10	6.18	0.744	-2.74	
12	7.67	0.799	4.46	
15	10.1	1.24	62.6	
18	13.8	1.24	62.1	
20	16.3	1.23	61.0	
21	17.5	2.07	170	>80% change in gas production rate, therefore α_2 [Step 5]
23	21.6	1.32	71.9	
25	24.3	1.08	41.1	
28	27.5	1.08	41.5	
30	29.7	0.806	5.36	
33	32.1	0.732	-4.39	
36	34.3	0.310	-59.4	
48	38.0	0.180	-76.5	
68	41.6	0.0970	-87.3	
92	43.9	0.0972	-87.3	
124	47.0	0.0554	-92.8	
142	48.0	-	-	

The colour scale was used as a visual aid to more easily identify the changes in gas production rate. For sample BH7A-A, the first decrease in gas production rate is calculated at t = 5 days, therefore the immediately preceding point, t = 3 days, is noted as the *alpha* point. The gas production rate at t = 21 days is found to be more than 80% than the rate at t = 3 days, therefore t = 21 days is noted as the new *alpha* point. No subsequent points show a gas production rate greater than at t = 21 days, therefore the lag phase was determined to end at t = 21 days, and the curve was fitted to all points with $t \geq 21$ days.

The effects of this modification are shown for samples BH16A-A and BH7A-A in Figure 5.1 and Figure 5.2, respectively. For sample BH16A-A, the R^2 value for the fit that includes all points was 0.9466, while the R^2 value for the fit that omits the lag phase was 0.9962, an increase

in goodness of fit of 5.2%. For sample BH7A-A, the R^2 value was 0.9742 for the all points fit and 0.9849 for the fit excluding the lag phase, an increase in goodness of fit of 1.1%.

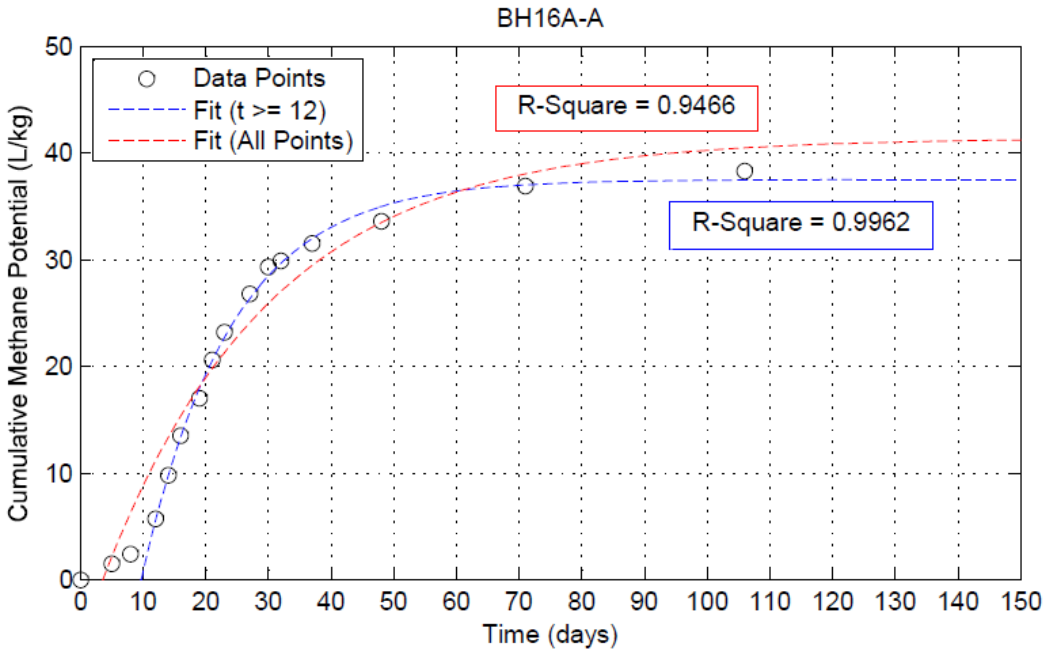


Figure 5.1: BMP results for sample BH16A-A showing the fitted curve using all points (red), the fitted curve omitting the lag phase (blue) and the difference in goodness of fit.

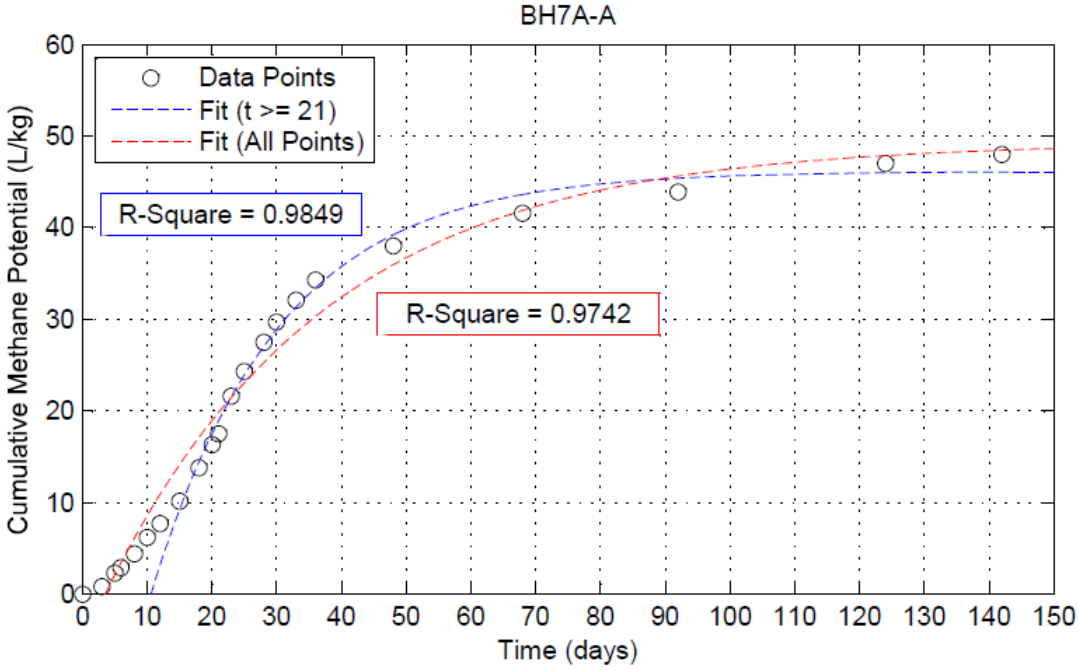


Figure 5.2: BMP results for sample BH7A-A showing the fitted curve using all points (red), the fitted curve omitting the lag phase (blue) and the difference in goodness of fit.

5.2.2. Multi-Stage Gas Production

The gas production curves of some samples exhibited multiple stages of gas production, indicated by humps in the curve. These samples required multiple fitted curves to accurately model the data. Sample BH9B-B provides an example of this, shown in Figure 5.3.

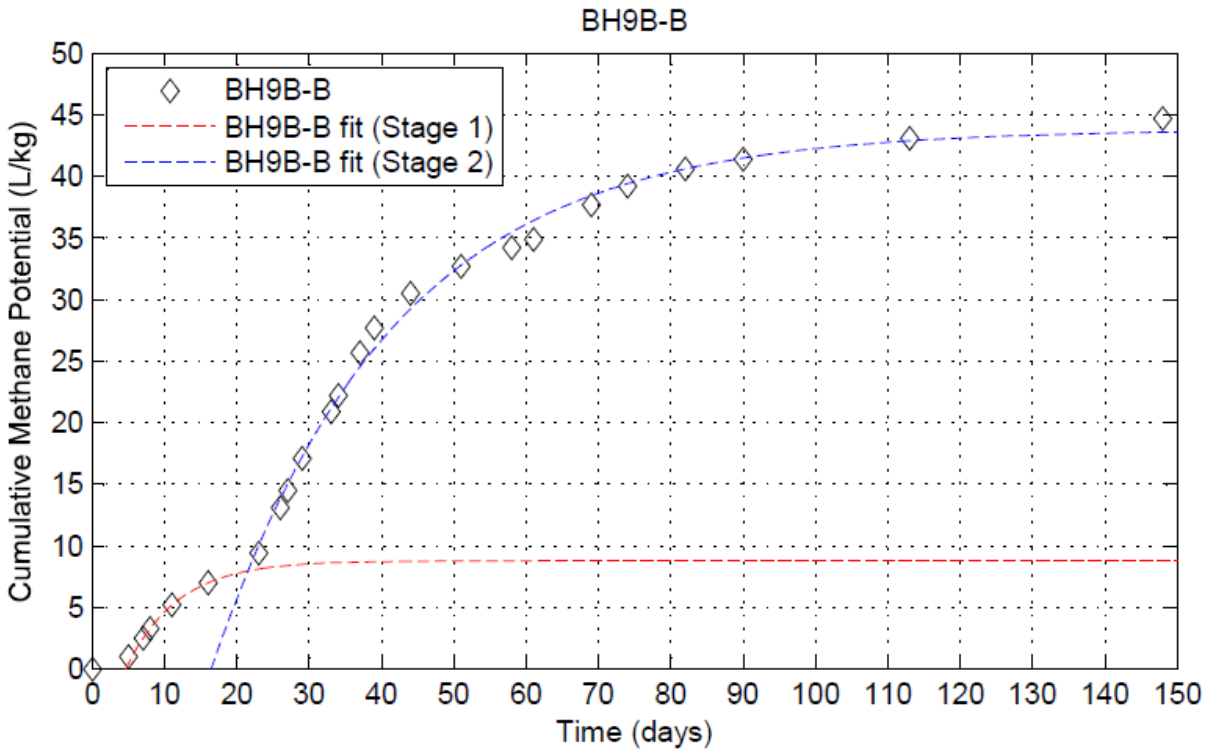


Figure 5.3: BMP results from sample BH9B-B showing dual-stage gas production and the fitted curves applied to each stage.

This multi-stage gas production was not always immediately obvious, and so it was necessary to develop an objective method for detecting multi-stage gas production using the BMP test data. The most basic indication of multi-stage gas production is a leveling out of the curve (reducing gas production rate), followed by a sudden increase in gas production rate. For this reason, the gas production rates between adjacent data points were once again used to determine the appropriate range of values to include in the fitted curves. Through a process of trial and error, the following two rules were developed to identify the beginning and end of multi-phase gas production stages:

1. For any gas production rate less than 0.45 L/kg/day (excluding the initial lag phase), if there is a subsequent increase in rate by at least 0.3 L/kg/day, then that indicates the end of one gas production stage, and the beginning of another.
2. For any gas production rate less than 0.25 L/kg/day (excluding the initial lag phase), if there is a subsequent increase in rate by at least 0.15 L/kg/day, then that also indicates the end of one gas production stage and the beginning of another.

If multiple stages of gas production were detected, the point before the beginning of the next stage at which the gas production rate was lowest was considered the point at which the stage ended. Each new production stage was preceded by its own pseudo lag phase, which was omitted from the fitted curves by the same process as the initial lag phase. The process of identifying multi-stage gas production is demonstrated in Table 5.2 using data from sample D-A, which exhibited three distinct phases of gas production.

By the process described in Section 5.2.1, the end of the initial lag phase was identified at t = 13 days. At t = 29 days it can be seen that the gas production rate drops below 0.45 L/kg/day, however, the next data point at t = 32 days has an even lower rate. Steps were saved by using t = 32 days as the baseline point to which the rest are compared, instead of t = 29 days, since the lower rate is more likely to trigger one of the above rules. At t = 36 days, there is an increase in rate of 0.37 L/kg/day, which is greater than 0.3 L/kg/day, indicating the end of the primary gas production stage at t = 32 days and the beginning of the secondary gas production phase at t = 36 days. Further on during the test, it can be seen that the gas production rate drops below 0.25 L/kg/day at t = 50 days. Once again, the following data point at t = 57 days shows an even lower rate, so steps are saved by using it as the baseline point to which the rest are compared. At t = 76 days the rate has reached 0.4 L/kg/day, an increase of more than 0.15 L/kg/day. This indicates the end of the secondary stage at t = 57 days and the beginning of a tertiary stage at t = 76 days. The three phases of gas production are plotted in Figure 5.4

Table 5.2: Example showing the methodology for the determination of the start points and end points of multiple gas production stages, using results from sample BH7A-A.

	t (days)	BMP (L/kg)	Gas Production Rate (L/kg/day)	Difference in Rate from t = 32 (L/kg/day)	Difference in rate from t = 57 (L/kg/day)	Notes
Stage 1	0	0.0	0.626	-	-	
	3	1.88	0.507	-	-	
	7	3.91	1.23	-	-	
	8	5.13	1.69	-	-	
	10	8.52	1.61	-	-	
	13	13.4	3.21	-	-	
	14	16.6	2.64	-	-	
	15	19.2	1.74	-	-	
	16	20.9	1.31	-	-	
	17	22.3	1.55	-	-	
	19	25.4	1.41	-	-	
	21	28.2	3.66	-	-	
	22	31.8	1.73	-	-	
	24	35.3	1.20	-	-	
	26	37.7	1.06	-	-	
	27	38.8	0.728	-	-	
	28	39.5	0.701	-	-	
	29	40.2	0.437	-	-	
	32	41.5	0.385	-	-	Gas production rate less than 0.45 L/kg/day
Stage 2	36	43.0	0.753	0.369	-	Gas production rate increase of more than 0.3 L/kg/day
	38	44.5	0.720	0.335	-	
	42	47.4	0.469	0.0837	-	
	50	51.2	0.219	-0.166	-	
	57	52.7	0.211	-0.174	-	Gas production rate less than 0.25 L/kg/day
Stage 3	62	53.8	0.253	-0.132	0.0424	
	76	57.3	0.400	0.0149	0.189	Gas production rate increase of more than 0.15 L/kg/day
	83	60.1	0.336	-0.0486	0.126	
	93	63.5	0.244	-0.140	0.0337	
	98	64.7	0.164	-0.221	-0.0466	
	111	66.8	0.0197	-0.365	-0.191	
	148	67.6	-	-	-	

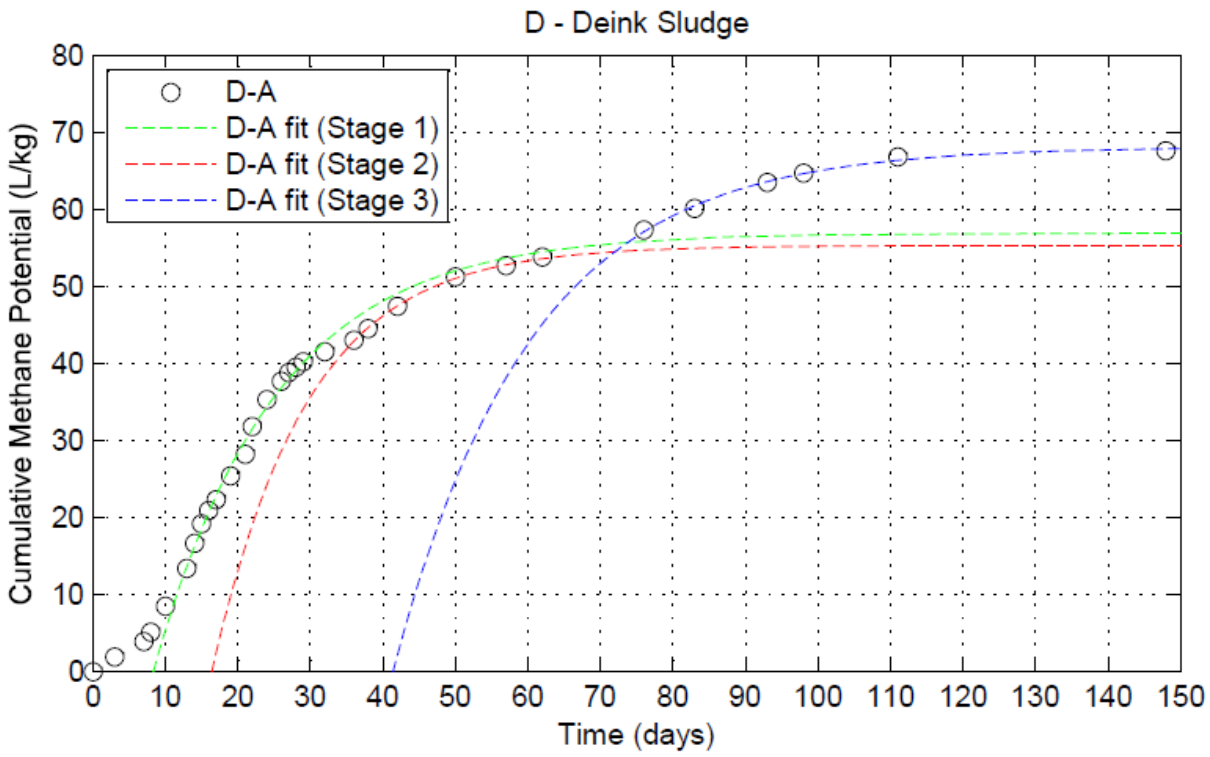


Figure 5.4: BMP results from sample D showing three stages of gas production and the fitted curves applied to each stage.

5.3. Predicting Multi-Stage Gas Production

Six samples showed multi-stage gas production behaviour in at least one of their replicate tests. These were BH2A, BH8A, BH9B, BH15A, S1, and D. Of the six, three showed it in all of the replicates (BH2A, BH8A, and BH9B), two showed it in two of three replicates (BH15A and S1), and one showed it in one of three replicates (D). Refer to Appendix B for the corresponding plots. The three samples that consistently showed multi-stage gas production across all replicates were also consistent in that the onset and rate of secondary gas production were in close agreement between replicate samples. The two replicates in each of BH15A and S1 that experienced multi-stage gas production did so at different times, and at different rates. From these results, it appears that if one replicate sample experiences multi-stage behaviour, it is likely that more will.

The testing was performed in batches of around 10 samples at a time, each with their three replicates. Inoculant for each batch of testing was gathered just prior to the beginning of testing, from the same source, at the same time. The six samples showing multi-stage behaviour are from four different test batches, separated by several months each. It is clear that the inoculant alone is

not the cause of multi-stage gas production, otherwise multi-stage behaviour would have been much more prevalent in some batches and/or completely absent from other test batches.

As described in Chapter 3, material from each sample was separated into two individual subsamples (A and B) during sample processing. As described in Chapter 4, a few tests were conducted on both A and B subsamples, and the two materials were found to provide essentially the same results. It is interesting then that sample BH2A showed clear multi-stage behaviour while BH2B did not. The two materials are essentially the same, and though different inoculants were used, it has already been determined that the inoculant alone does not govern multi-stage behaviour. Some other possible causes of multi-stage behaviour are as follows:

- minor differences in the testing environment such as airflow and/or temperature gradients within the incubator;
- small differences in the handling of test samples such as time spent out of the incubator while taking readings, or slightly less vigorous agitation at test set-up;
- some unknown interaction between specific test materials and specific inoculants; and,
- contamination by some outside organism during test set-up.

As it is, the cause of multi-stage gas production behaviour remains unknown. Fortunately, the results show that the occurrence of multi-stage gas production does not prevent a quantitative, rigorous, and systematic analysis to determine the ultimate BMP value. The average BMP_{ult} value for BH2A of 26.3 L/kg was in close agreement with the avaerage from BH2B of 27.0 L/kg, and the coeffiecients of variation for samples BH15A, S1, and D averaged to 1.64%, which is less than the average of 1.94% for the samples that didn't show multi-phase gas production.

5.4. BMP_{60}

Many BMP methods in the literature specify a maximum incubation time. This is desirable because waiting for the test to reach completion can take as long as six months or more, which not only ties up lab space for that entire period, but may also be prohibitive to time sensitive projects. The results of this study show that the rate of gas production drops sharply well before the end of the test is reached, with most of the gas being produced in that initial high production rate phase. The results of a 50, 60, or 75 day test therefore are still valuable in that a technician will likely know in that time whether the ultimate BMP value of a sample will be closer to 15 L/kg or 50 L/kg,

but the fact remains that these methods are underestimating the ultimate BMP value. With this in mind, this study set out to determine how accurately ultimate BMP values could be predicted from incomplete data using the first-order decay model described in Section 5.2. If successful, reasonably accurate ultimate BMP results could be obtained without the expense of an overly long test duration.

5.4.1. Fitting Curves for BMP_{60}

The specific amount of time chosen for this analysis is essentially arbitrary. A period of 60 days was chosen simply because that was the length of the test method used by researchers at North Carolina State University, to whom certain samples from this study were sent in order to compare BMP methods. Curves were fit to all data points up to approximately 60 days (excluding the lag phase, as described in Section 5.2.1). Since measurements were taken only when sufficient gas had been produced for a reading, data points were not available at exactly 60 days for all samples. In these cases, the data point closest to 60 days was used as the last point included in the fit. An example of the fit using only the data up to 60 days is given for sample BH3A in Figure 5.5. BMP_{60} plots for all samples are provided in Appendix E. Some tests were terminated before 60 days, because they were deemed to have reached completion. No BMP_{60} curves were fitted to these samples, and they have not been included in the following analyses.

In the example shown in Figure 5.5 there is only one data point available past 60 days, and thus this fit is very similar to the one using all data points (see Appendix B). For samples tested in this study, it was typical to have only one to three data points with t values greater than 60 days, due to the greatly reduced gas production rate by that time. For those few samples with four or more points beyond 60 days, the accuracy of the BMP_{60} fit was less predictable. Some of these samples maintained relatively good agreement between the BMP and BMP_{60} data, but most were noticeably underestimated as shown in Figure 5.6 using sample BH6A.

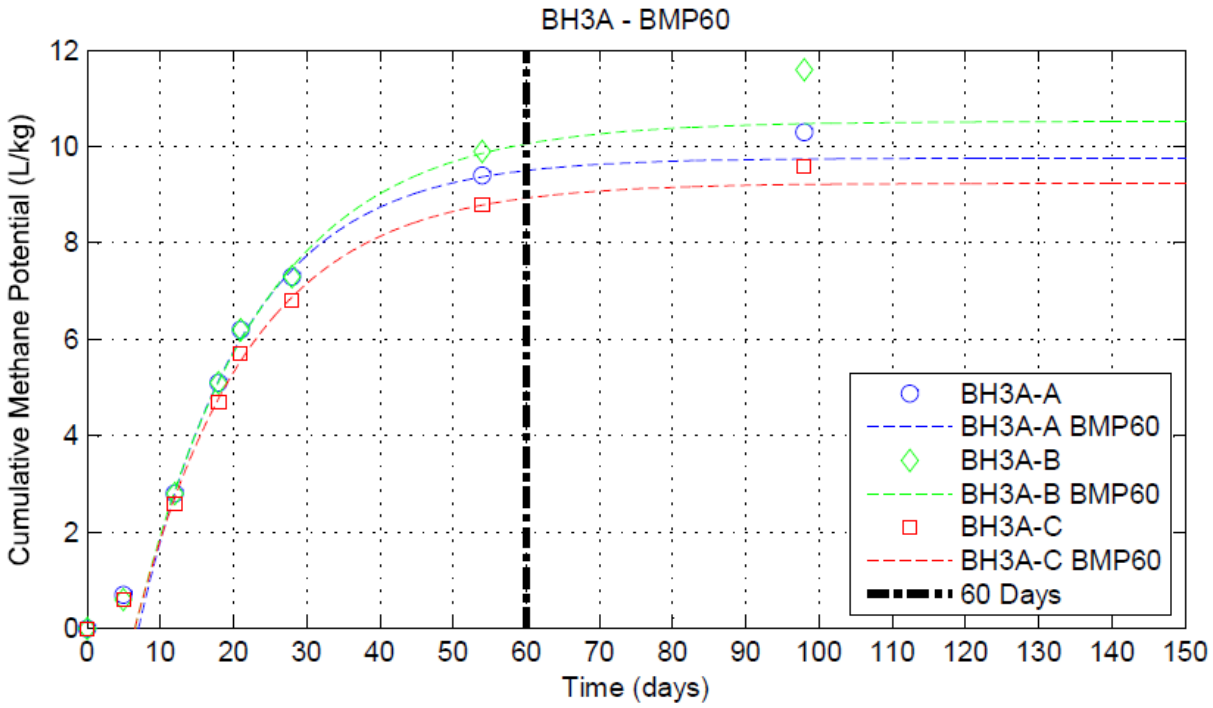


Figure 5.5: BMP results for the three replicate samples of BH3A, showing curves fitted using only data up to 60 days.

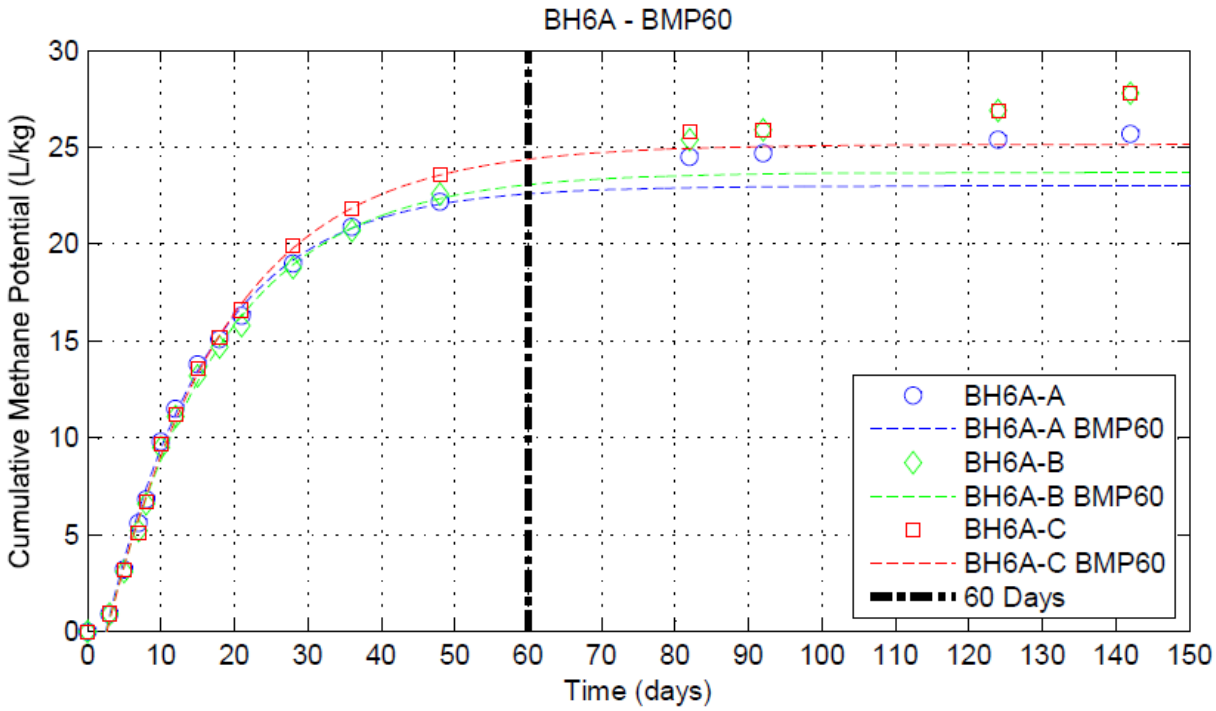


Figure 5.6: BMP results for the three replicate samples of BH6A, showing curves fitted using only data up to 60 days.

5.4.2. Comparing BMP and BMP_{60}

Table 5.3 provides a comparison between the ultimate BMP values determined using all data and the predicted ultimate BMP values using data only up to 60 days (BMP_{ult60}). BMP_{ult60} values were not calculated for samples that reached their ultimate methane potential in less than 60 days.

Table 5.3: Comparison of BMP_{ult} values, to BMP_{ult60} values.

Sample	Average BMP_{ult} (L/kg)	Average BMP_{ult60} (L/kg)	Percent Error
BH1A	32.2	31.9	-1.20
BH2A	26.3	28.1	6.83
BH2B	27.0	24.9	-7.67
BH3A	10.5	9.85	-5.85
BH4A	6.71	6.95	3.51
BH5A	12.7	13.7	7.47
BH6A	26.1	24.0	-8.17
BH7A	45.7	44.4	-2.80
BH8A	22.3	21.3	-4.66
BH9B	41.0	36.6	-10.75
BH10A	29.2	28.7	-1.80
BH11A	24.7	26.7	8.06
BH12A	13.3	12.5	-5.67
BH13A	38.8	39.7	2.24
BH14A	5.56	5.06	-8.97
BH15A	17.0	15.6	-8.04
BH16A	38.1	37.0	-2.93
BH17A	15.4	14.5	-6.05
S1	62.6	59.6	-4.76
S2	6.94	5.72	-17.5
S3	138	156	12.8
S4	89.1	102	14.2
S5	3.41	3.00	-11.9
S6	4.07	3.38	-17.0
S7	2.76	2.38	-13.7
S8	9.62	9.72	1.00
S9	5.53	5.99	8.31
S10	18.3	14.2	-22.5
D	63.5	58.2	-8.37
W	68.4	67.1	-1.97
CS	60.1	57.2	-4.74
CS/MSW	49.2	47.2	-4.12
		Average	-3.65
		Standard Deviation	8.60

Of the 32 samples analyzed, 72% yielded BMP_{ult60} results lower than the BMP_{ult} results. This indicates a tendency for the BMP_{ult60} to underestimate BMP_{ult}, but with an average percent error between the two values of only -3.65% there is still fairly good agreement. Figure 5.7 shows how the BMP_{ult} results plot against BMP_{ult60} results. The resulting fit is very good, with an R^2 value of 0.9877, and narrow 95% prediction bounds. This makes BMP_{ult60} a good way to predict of BMP_{ult} in a reasonably short amount of time.

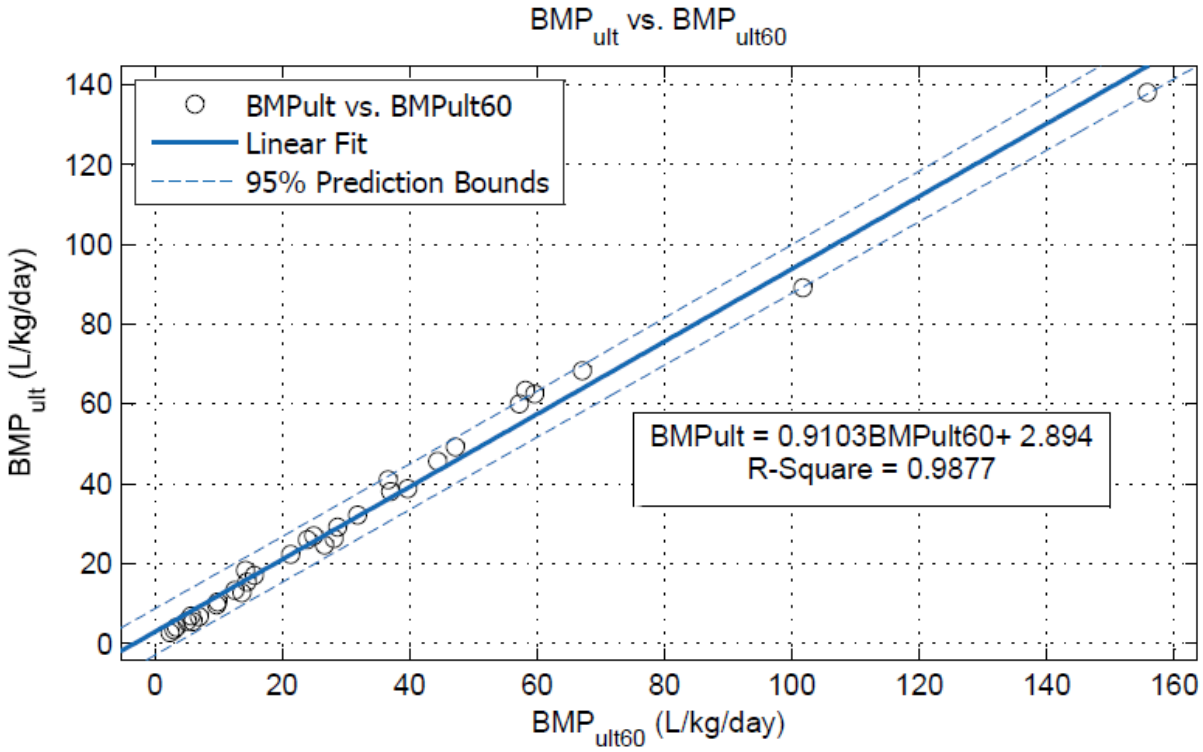


Figure 5.7: Plot showing the relationship between BMP_{ult} values and BMP_{ult60} values.

5.4.3. Multi-Stage Gas Production and the BMP_{60}

Using the method described in Section 5.2.2, multi-stage gas production can be detected and accounted for in the BMP_{60} if it occurs earlier than 60 days into the test, as it did in most cases. Where multi-stage behaviour was detected before 60 days, two fitted curves were developed to analyze the BMP_{60}, one using all of the data up to 60 days (or the closest data point to 60 days, excluding the lag phase), and one using only the data between the onset of the second stage and 60 days. Where applicable, both of these curves are provided in the plots in Appendix E. An example is shown here, in Figure 5.8, for sample BH8A.

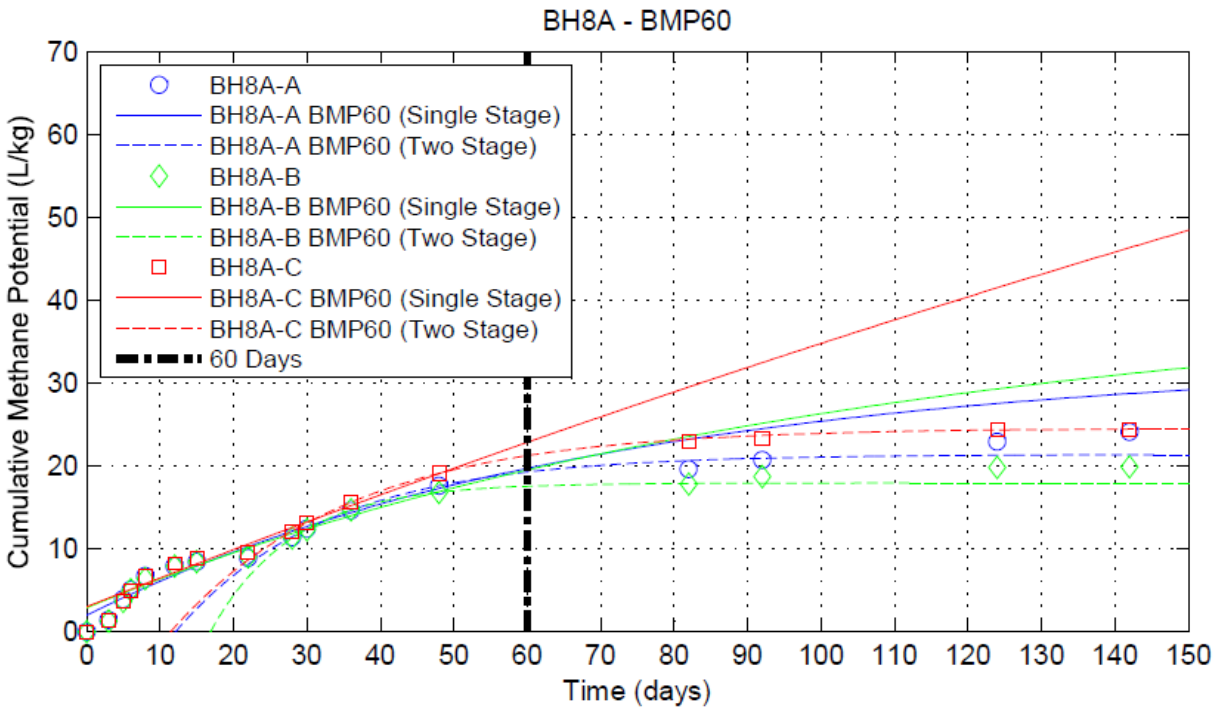


Figure 5.8: BMP results for the three replicate samples of BH8A, showing curves fitted using only data up to 60 days. Curves shown in solid lines were fitted using all data up to sixty days. Curves shown in dotted lines were fitted only to that data (up to 60 days) which represents the second stage of gas production.

In this example it can be clearly seen that the BMP_{60} curves that account for two-stage gas production provide a much better estimation of BMP_{ult} than those that don't. This was the case for all but one of the 13 total tests that experienced multi-stage gas production; therefore these were the curves used for the purposes of analysis.

5.5. Correlation to LOI

Plots of BMP vs. LOI, at each temperature tested are shown in Figure 5.9 through Figure 5.14. Except for a few outliers, the data appears to follow a linear pattern. The best fit was achieved at 250°C, with an R^2 value of 0.75. At temperatures above 250°C the R^2 value drops significantly and remains around 0.54. This seems to be because of two outliers (S9, and A) that appear at 450°C. At temperatures above 450°C all other changes are minor, and appear fairly uniform across all samples. These outliers are highlighted in Figure 5.15.

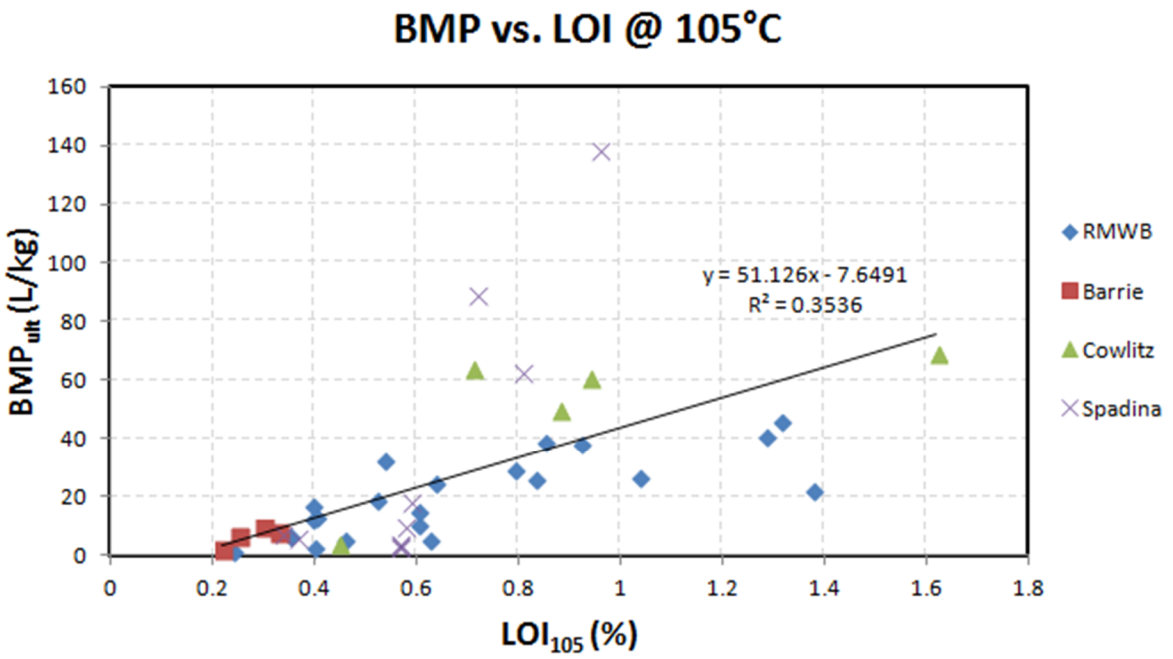


Figure 5.9: Plot of average BMP_{ult} vs. average LOI_{105} for all samples.

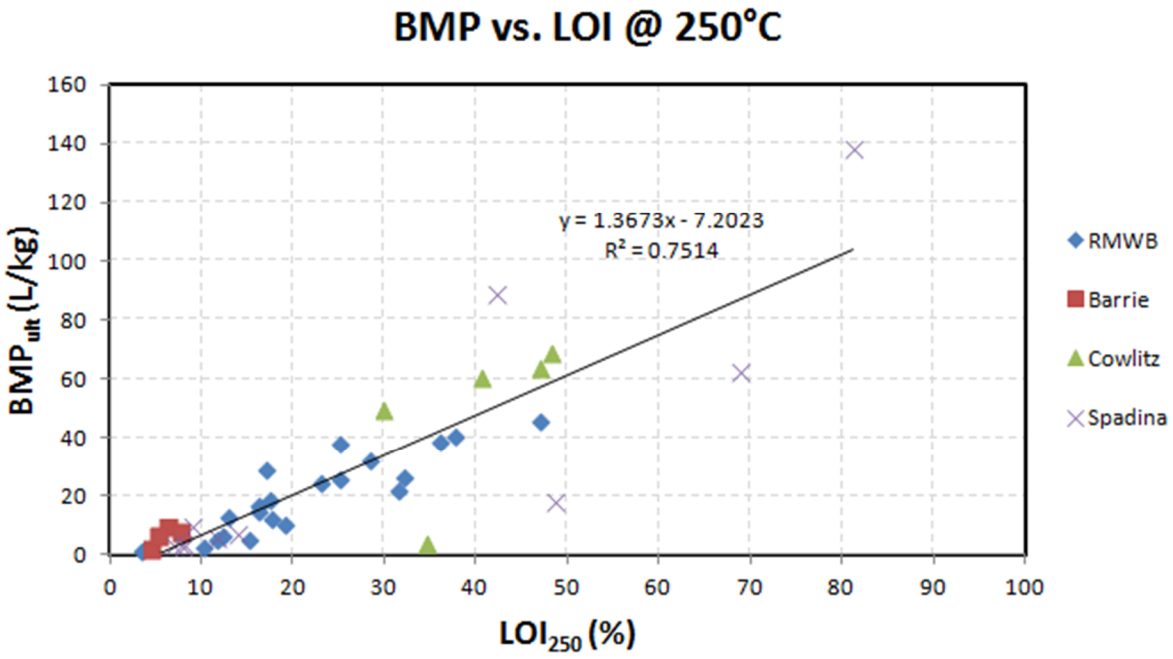


Figure 5.10: Plot of average BMP_{ult} vs. average LOI_{250} for all samples .

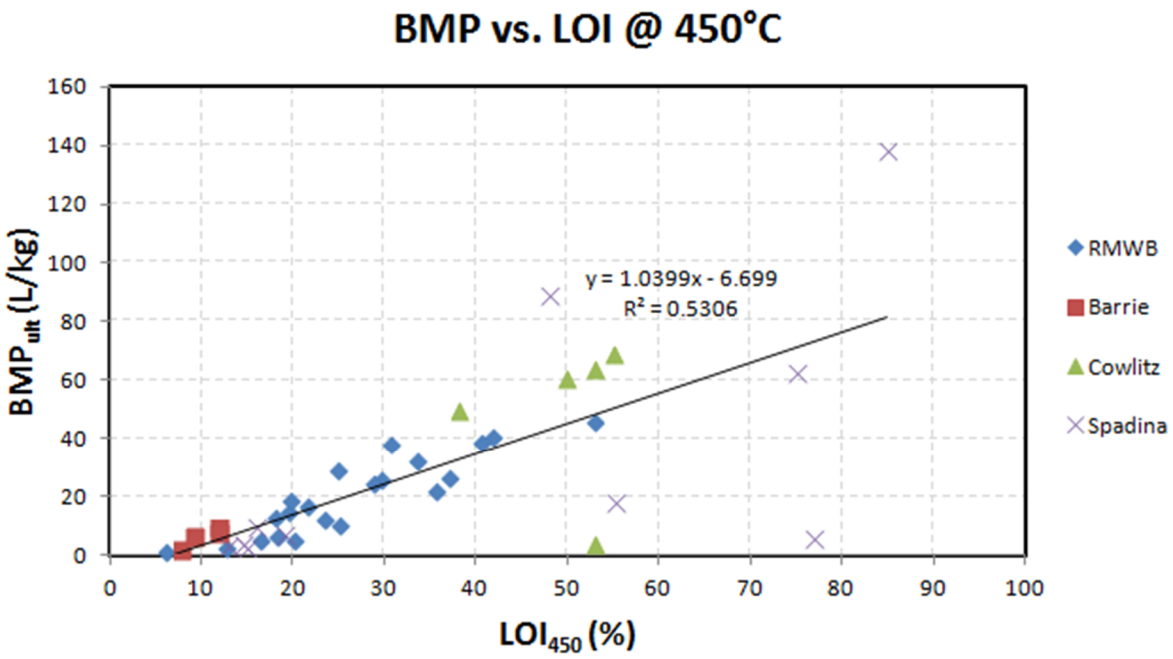


Figure 5.11: Plot of average BMP_{ult} vs. average LOI_{450} for all samples .

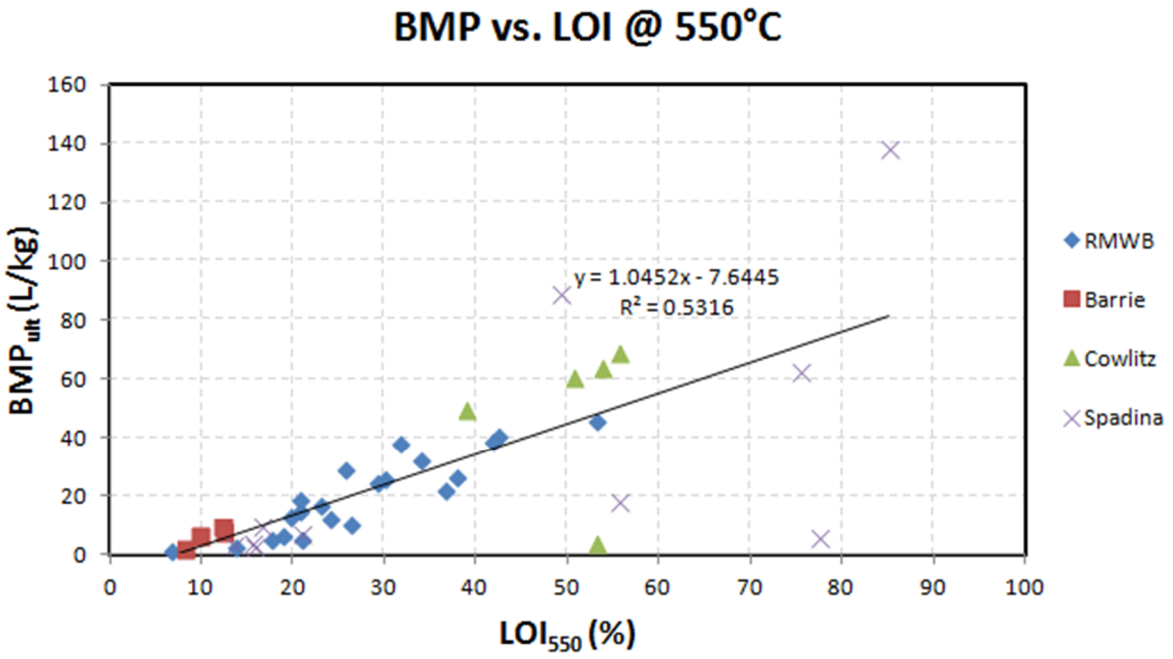


Figure 5.12: Plot of average BMP_{ult} vs. average LOI_{550} for all samples.

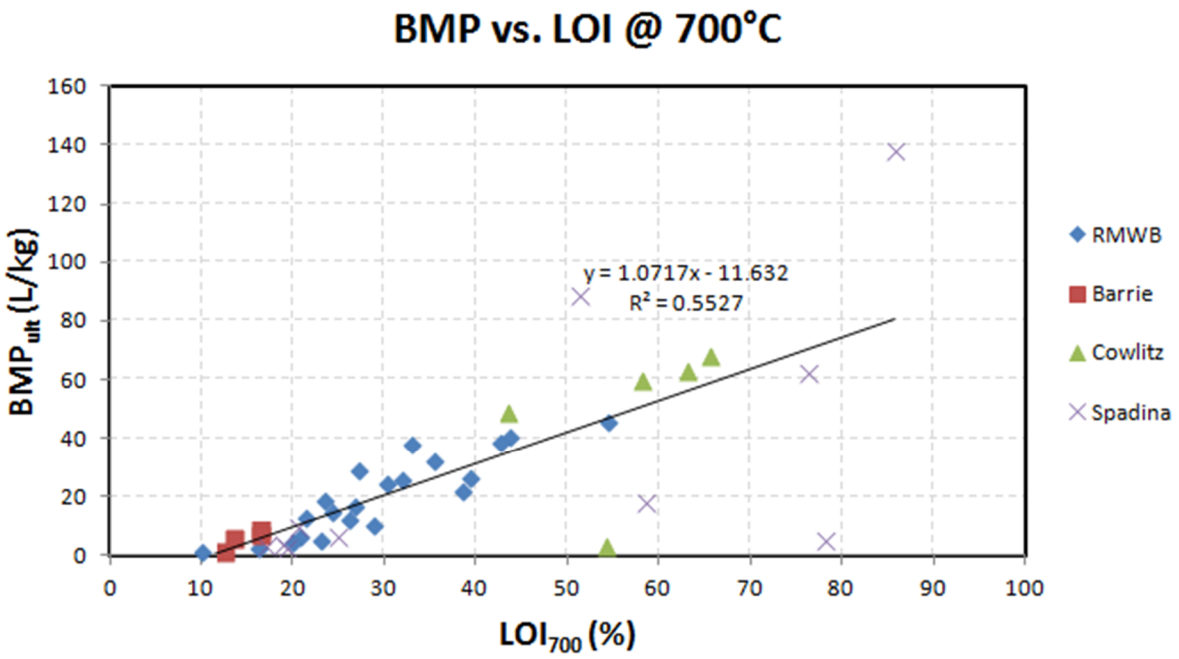


Figure 5.13: Plot of average BMP_{ult} vs. average LOI_{700} for all samples.

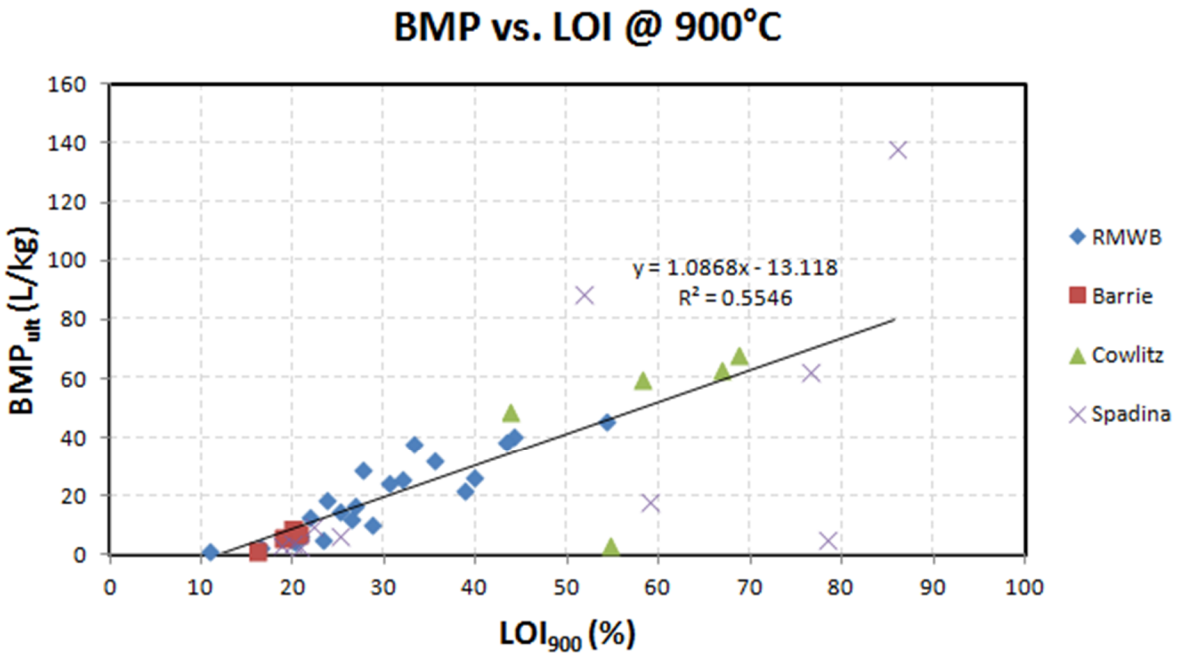


Figure 5.14: Plot of average BMP_{ult} vs. average LOI_{900} for all samples.

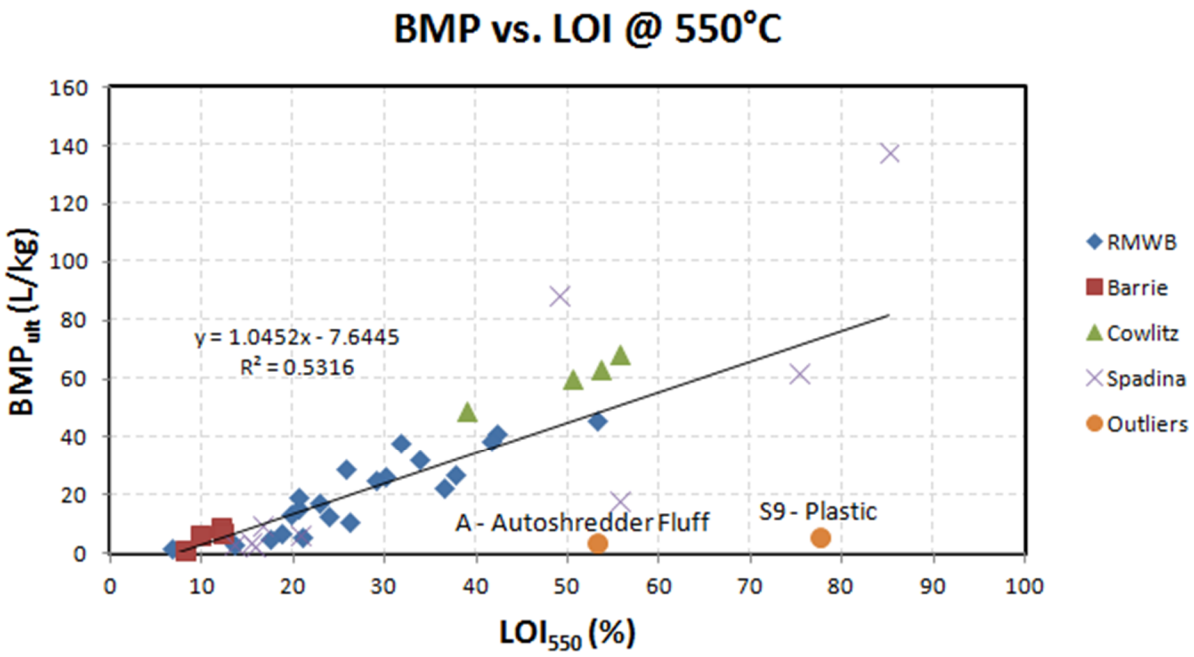


Figure 5.15: Plot of average BMP_{ult} vs. average LOI_{550} for all samples, highlighting outliers S9 and A.

The statistical analysis software SPSS (IBM Corporation, Armonk, NY, USA) was used to perform multiple linear regression analyses to determine if any manipulation of the data would result in a better model. Various combinations of the LOI values at different temperatures were input. While some combinations did result in models that produced a good fit, very few of the coefficients of the independent variables of these models were statistically different from zero, indicating that those variables likely were not contributing to the model.

One common property of the outliers S9 and A is an extremely high plastic content. Plastic is a non-biodegradable organic material. Therefore, it will burn off during an LOI test, but will not contribute to the BMP. Since the two major outliers both appeared between 250°C and 450°C a new variable ($LOI_{450-250}$) was created by finding the difference in LOI between these two temperatures. It was thought that this variable might serve to represent plastic content in the samples. By plotting BMP against LOI_{550} minus $LOI_{450-250}$ the results plotted in Figure 5.16 can be achieved.

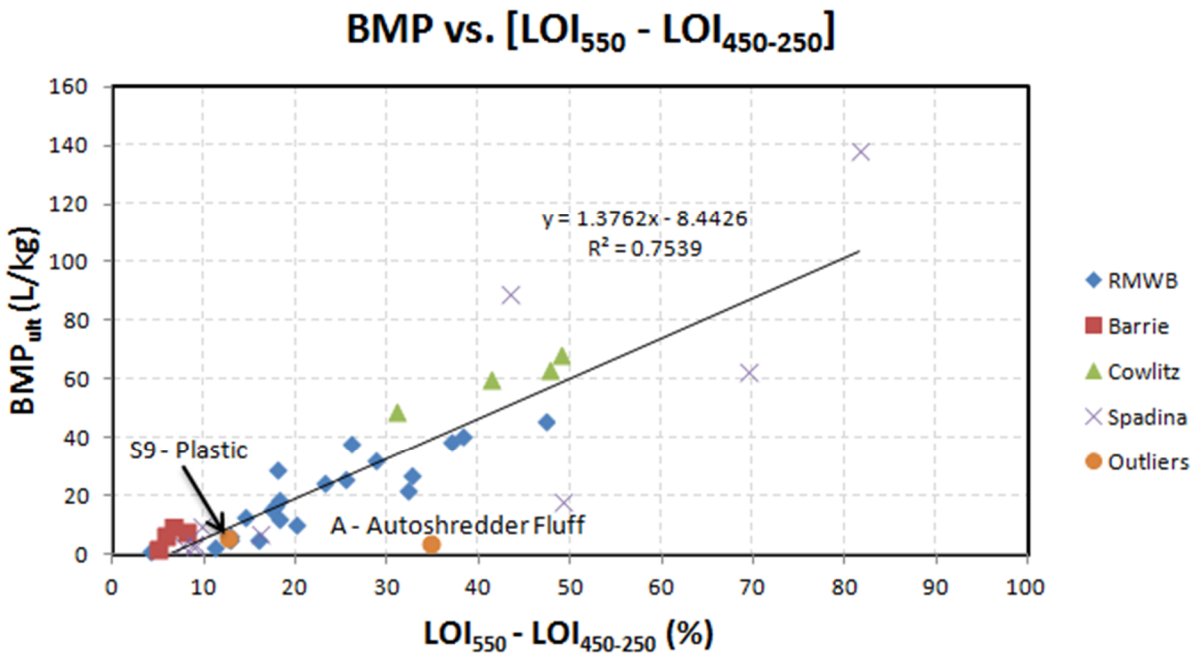


Figure 5.16: Plot of average BMP_{ult} vs. LOI_{550} - (LOI_{450} - LOI_{250}) for all samples, highlighting previous outliers S9 and A.

Comparing against Figure 5.15 it can be seen that the two outliers have shifted to the left dramatically, while the other points have remained relatively close to their original positions. This supports the claim that the difference in LOI between 250°C and 450°C is an indicator of the plastic content.

The composition of MSW in North America tends to be fairly consistent and plastic accounts for approximately 8-13% of it (Karak et al. 2012). The difference in LOI between 250°C and 450°C for most samples was actually below this range, so the value likely underestimates the plastic content. For samples S9 and A however, the value was 65.16% and 18.57% respectively; well above the range for normal MSW. This allows us to conclude that any MSW sample with LOI450 – LOI250 > 13% is likely an outlier and will not conform to the correlation.

Other variables were created to represent the differences between LOI at adjacent temperature values tested (i.e. $LOI_{550-450}$, $LOI_{700-550}$, etc.). These variables were also input to SPSS and various combinations tested for multiple linear regression. No statistically significant relationships could be found other than between $LOI_{450-250}$ and each of the LOI values above 450°C, individually. The statistical significance of each of these relationships was extremely similar. The best relationship, in terms of statistical significance, was found using LOI_{900}. The relationship is as follows:

$$\boldsymbol{BMP_{ult} = -13.487 - 1.490(LOI_{450-250}) + 1.394(LOI_{900})} \quad \text{(Equation 5.2)}$$

This relationship predicted BMPult statistically significantly, $F(2,37) = 61.769$, $p < 0.0005$, $R2 = 0.770$. Both variables contributed significantly to the prediction, $p < 0.05$. This relationship provides only a marginally better fit than the relationship between BMPult and LOI250, suggesting that good BMPult results can be predicted by simply measuring the LOI at 250°C, before the plastics are burnt off.

Using the relationship shown in Figure 5.16, the ultimate BMP predicted for all samples was within ±40.9 L/kg of the values determined by the modified method. This error is large, and therefore confidence in the precision of this test is not high when considering only individual samples. However, the average error of all samples is only -0.130 L/kg; showing that there is very little bias to this method, and precision can be achieved when characterizing a large mass of waste by testing a sufficient number of samples. Confidence in the precision of individual test samples is much higher when considering only the samples representing homogenized waste, (i.e. excluding S1, S3, S4, S9, S10, and the Cowlitz samples). In this scenario the error is reduced to ±13.4 L/kg.

The temperature intervals used in this study were rather large, as it was unknown what might contribute to the relationship at the beginning of the study. Now that it has been shown that there is little contribution to BMP_{ult} by substances with ignition temperatures greater than about 450°C, a better relationship might be found by repeating the analysis with tighter temperature intervals between 105°C and 450°C. Using the data in **Error! Reference source not found.**, it may be possible to identify the proportions of more specific organic compounds (both biodegradable and non-biodegradable), allowing for more precise control of such variables within the multiple regression relationship. Temperature intervals that identified wood content and paper content would be of particular use, as can be seen in Figure 5.17, which is a replica of Figure 5.16 with the mainly wood and paper Spadina samples highlighted. This figure appears to indicate that wood contributes more to LOI than BMP, while paper contributes more to BMP than LOI. This is supported by the fact that wood contains lignin, which is less biodegradable than cellulose, as well as physically inhibits the degradation of some of the cellulose (see Section 2.5.4). Unfortunately, the variability of different wood and paper products (density, treatment, etc.) make

the identification of one specific ignition temperature practically impossible, with ignition temperatures reported in the literature ranging from 200°C to 510°C (Babrauskas, 2001).

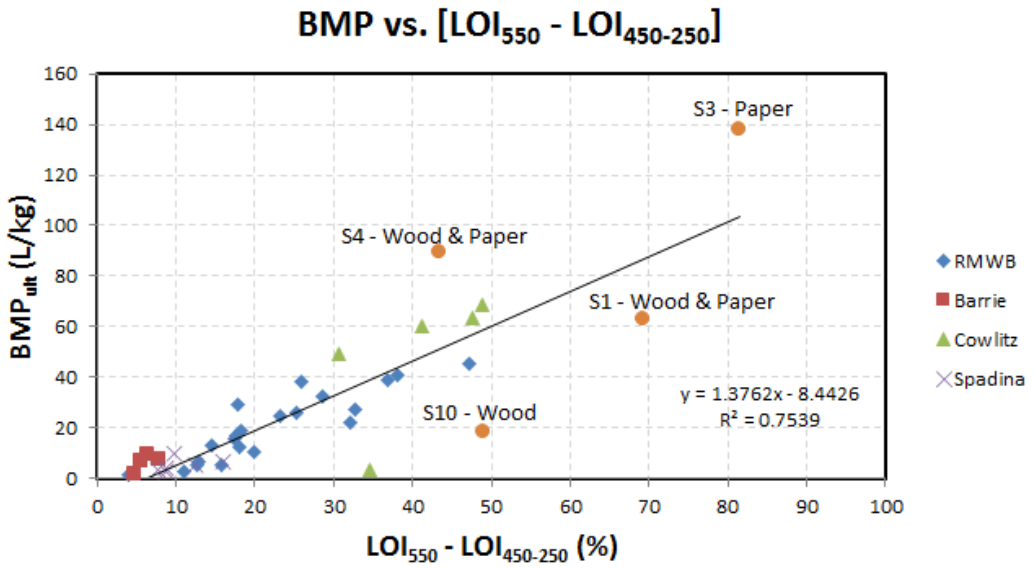


Figure 5.17: Plot of average BMP_{ult} vs. LOI_{550} - (LOI_{450} - LOI_{250}) for all samples, highlighting samples with high wood and paper content .

5.6. Correlation to Specific Gravity

Figure 5.18 presents the ultimate BMP data plotted against specific gravity (G_s) test results.

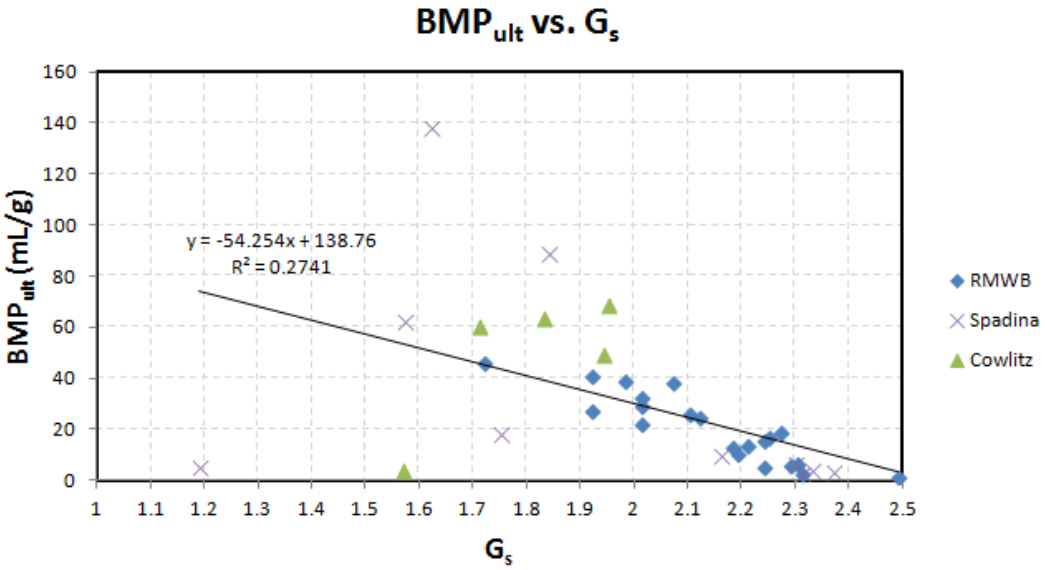


Figure 5.18: Plot of average BMP_{ult} vs. Specific Gravity for RMWB, Spadina, and Cowlitz samples.

Initially, there does not appear to be a good relationship between BMP and specific gravity. Similar to the LOI however, specific gravity results are highly dependent on organic content. If the two outlier samples with high plastic contents are omitted from the regression, the relationship becomes much better, however with an R^2 value of 0.65 it is weaker than the correlation to LOI. This is shown in Figure 5.19.

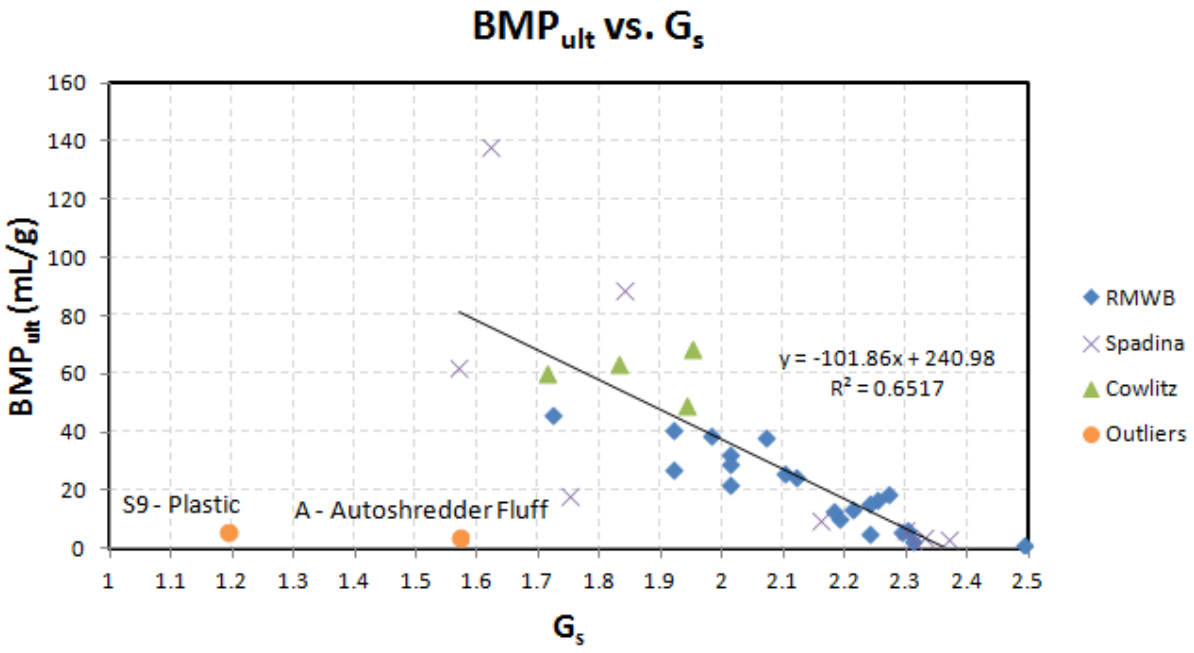


Figure 5.19: Plot of average BMP_{ult} vs. Specific Gravity for RMWB, Spadina, and Cowlitz samples, with outliers S9 and A highlighted and omitted from trendline.

Using the relationship shown in Figure 5.19, the maximum error in predicted ultimate BMP value is 114 L/kg, and the minimum is -62.1 L/kg. Average error for all samples is 5.18 L/kg. These errors are too large for this correlation to be of use in determining the ultimate BMP value of an individual sample, and the method shows a clear bias towards positive error on average. Greater precision is achieved when considering only homogenized waste samples. In this scenario the maximum and minimum error become 20.1 L/kg and -14.1 L/kg, respectively. This error is still greater than what can be achieved by the LOI correlation however, and the bias towards positive error is still apparent.

Attempts were made to include the specific gravity data in a multiple linear regression with LOI data in order to better predict the ultimate BMP. No linear regression model including specific gravity data was found that could provide an improvement over the models already described,

using LOI data alone. It was also determined that specific gravity did not perform better at identifying outliers than did the combination of LOI250 and LOI450 described in Section 5.5. Because of this, it was concluded that the specific gravity does not provide any useful data for determining ultimate methane potentials.

5.7. Correlation to BCP

Only a relatively small amount of data was gathered from the BCP test, but what there is shows some promising results. Similar to how the BMP_{ult} was predicted from BMP data at 60 days in Section 5.4.2, the BCP at 30 days (BCP_{30}) and at 50 days (BCP_{50}) was plotted against the ultimate BMP values. The result is shown in Figure 5.20.

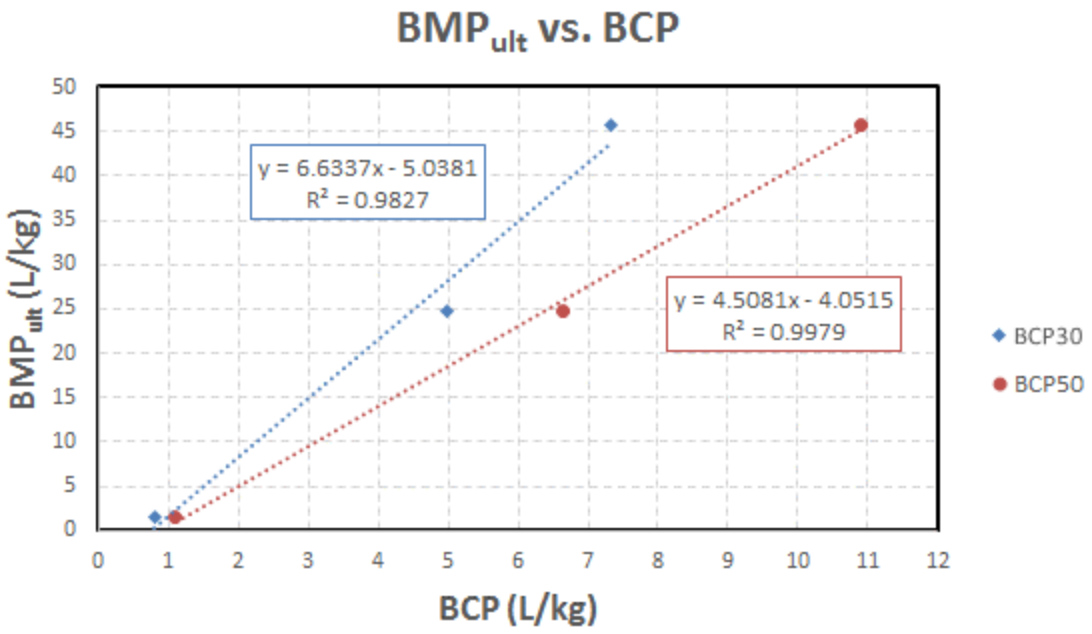


Figure 5.20: Plot of BMP_{ult} values vs. BCP values after 30 days (blue) and 50 days (red).

Even after only 30 days, the BCP data shows a good correlation to BMP_{ult}, suggesting that the BCP test may indeed work as a quick and accurate method for determining BMP_{ult}. However, with only 3 data points, the true quality of the correlation may not be as high as it appears here. Further study is needed to confirm this correlation.

5.8. Summary

This chapter presented a detailed analysis of the results reported in Chapter 4. Methane production was shown to follow a first-order decay model; the parameters of which could be fairly accurately determined 60 days into the test. The LOI at 250ºC was shown to be a practical way of quickly estimating ultimate BMP. It was also shown that LOI values at higher temperatures could be used to estimate ultimate BMP values with similar accuracy, provided variations in plastic content were accounted for. Through the process of performing sequential LOI tests on the same samples, it was shown that the effect of plastic content on the BMP LOI correlation at higher temperatures could be accounted for to some degree. A relationship between specific gravity and ultimate BMP was also found, though the correlation was much weaker, and it first requires the values from the sequential LOI tests to identify and exclude outliers. Preliminary investigation of the BCP test showed that it may be possible to predict ultimate BMP values from a BCP test in as little as 30 days, but this conclusion is based on a very small data set and further study is required to confirm the relationship.

Chapter 6 Conclusions and Recommendations

6.1. Introduction

This chapter summarizes and critiques the work that was presented in this thesis. The conclusions that can be drawn from this study are highlighted, and recommendations for performing BMP testing are given. Possible limitations of the study and its applicability are also discussed. Lastly, suggestions for future research based on this work are offered.

6.2. Summary

The objective of this study was to provide alternative methods for determining the BMP of MSW, that would improve upon existing methods in one or more of these three areas:

1. Time requirement;
2. Repeatability; and,
3. Need for specialized equipment.

Three options were explored for achieving these objectives:

1. A modified BMP method;
2. Correlation of BMP to results to LOI and specific gravity; and,
3. Development of an aerobic analogue to the BMP test, dubbed the BCP.

Chapter 2 contained background information and literature review related to BMP tests. Chapter 3 described the materials, equipment, and procedures used in the study. The experimental results were presented in Chapter 4. Detailed analysis of the experimental results was carried out in Chapter 5.

6.3. Conclusions

6.3.1. Modified BMP Method

The modified BMP method developed for this study used a sample mass much greater than normal for BMP testing. The result of this was much larger volumes of gas. Collecting and analyzing gas samples repeatedly, and in large quantities, meant that any random errors in the data tended to average out over the course of the test. Using large samples to produce a relatively large volume of gas enabled the use of less precise, but more economical and easier to use, testing equipment. In comparison with other methodologies for this test that use a much smaller sample (up to 200 times less) and very precise analysis of produced gas, it can be demonstrated that the proposed sample method is as accurate, or more so, in practical terms. The method presented here gave results with a similar, or slightly better repeatability, and higher ultimate methane potentials. This has been shown to be the case based on inter-lab testing of replicate samples at NCSU. The method was used on a wide range of waste materials, proving reliable in most cases. Overall, the BMP test method described in this study provides an accurate and cost effective alternative. The time required to complete the test by this method can be significant, but it was found that the ultimate methane potential could be accurately estimated at 95% confidence after only 60 days of testing.

6.3.2. Loss on Ignition

Sequential LOI analysis was carried out on each of the samples tested. Temperatures included in the progression were 105ºC, 250ºC, 450ºC, 550ºC, 700ºC, and 900ºC. When plotted against ultimate BMP values, it was found that a linear relationship existed between LOI and BMP at all temperatures from 250ºC and above. It was found that the samples with higher than average plastic content distorted the relationship, but this could be accounted for by taking the difference between the LOI at 450°C and at 250°C as a representation of plastic content.

6.3.3. Specific Gravity

The specific gravity of each sample was tested. ASTM D854 was found to be a reliable method of determining specific gravity of waste samples, despite the occasional presence of absorptive, or floating particles. A rough linear relationship was evident between BMP and specific gravity. Similar to the LOI correlation, this correlation was also found to be distorted by waste samples

containing higher than average plastic content. The specific gravity was not found to provide any useful data for the prediction of ultimate BMP values; neither as a predictor of outliers, nor as an addition to a multiple linear regression between BMP_{ult} and LOI.

6.3.4. BCP Trial

The initial attempt at developing an aerobic analogue to the BMP test showed that while the CO_2 production rate and cumulative value produced did vary as expected, the test as carried out was insufficiently more rapid than the BMP. Analysis of the partial BCP results showed that there may be a good correlation between early BCP results BMP_{ult}, but there is insufficient data to reliably confirm this. There exists several possibilities for increasing the rate of degradation, and with future research it may yet be viable to take the method to completion in a relatively short time.

6.4. Limitations

6.4.1. Modified BMP Method

Each BMP test provides the methane potential for only a small portion of any landfill. To get an accurate estimation of methane potential for an entire landfill requires many individual samples. A duration of up to 6 months for a single test means that either a very large incubator, or a very long time is needed for comprehensive BMP testing. Inhibition is another problem. Due to the variability of waste, and the impracticality of a rigidly controlled inoculum source, chemical inhibition remains unpredictable.

6.4.2. LOI Correlation

The correlation between BMP and LOI is dependent on MSW samples containing "normal" ratios of waste components. The relationship described in this thesis almost certainly won't apply in regions where waste disposal attitudes and practices are significantly different than they currently are in the average North American region. Neither will it apply to waste samples that have been processed in a way that may significantly affect those ratios. Even given these conditions, the correlation is a rough one. Assuming the waste sample conforms to the requirements of the correlation, expected errors may be in the range of ±13.4 L/kg. If greater precision is required, the modified BMP method presented in this thesis should be used instead.

6.4.3. Specific Gravity Correlation

The correlation found between BMP and specific gravity was much weaker than that found between BMP and LOI. As a stand-alone analysis it is not likely to provide very good information and should instead be employed as an indication of expected BMP values that is complimentary to that determined from LOI testing. Like the correlation between BMP and LOI, the correlation between BMP and specific gravity is also dependent on "normal" ratios of waste components. Assuming the requirements for the application of the correlation are met, expected errors may be in the range of ±20.1 L/kg, with a bias towards positive error.

6.5. Recommendations

When performing BMP tests on MSW, this research shows that there is considerable benefit to using large sample masses. The test is less delicate, yet the precision of the results is comparable to more traditional methods. It has been found that most experimental factors do not affect the extent of degradation, but many can influence the rate (Raposo et al. 2011). The following lists several ways it is possible to maximize the rate of degradation without effecting extent:

- Temperature between 34-38ºC (Barlaz et al. 1990);
- Larger inoculum to substrate ratio (Raposo et al. 2011);
- Constant agitation (Raposo et al. 2011); and,
- Addition of other waste products (Białowiec and Templin 2011);

When attempting to estimate BMP from LOI, it is recommended that the waste is first characterized according to composition. If an analysis of the LOI at 250°C minus the LOI at 450°C yields a result greater than 13% that sample is likely an outlier that will not conform to the correlation.

6.6. Proposed Methods

This section presents step-by-step summaries of the various methods proposed for measuring BMP_{ult} based on this research.

6.6.1. LOI Method

The following is the proposed step-by-step method for estimating BMP_{ult} of a MSW sample based on sequential LOI results:

1. Dry out the bulk sample in an oven at 60°C.
2. Shred the sample to a maximum particle diameter of no greater than 3 mm.
3. Perform a sequential LOI test on three replicate samples as follows:
    a. Add at least 50 g of sample to three individual crucibles with known tare masses.
    b. Put the crucibles in an oven/furnace at 60°C, 250°C, 450°C, and 900°C for no less than 4 hours at each temperature, weighing the crucibles after each temperature interval.
4. Calculate the average LOI at 250°C, 450°C, and 550°C using the LOI at 60°C as a correction for residual water content.
5. Estimate BMP_{ult} using the relationship shown in Figure 5.16.

6.6.2. BMP_{60} Method

The following is the proposed step-by-step method for estimating BMP_{ult} from a 60 day BMP method:

1. Dry out the bulk sample in an oven at 60°C.
2. Shred the sample to a maximum particle diameter of no greater than 3 mm.
3. Anaerobically digest the sample in three replicate tests as follows:
    a. Add 200 g of sample, 1 L of distilled water, and 200 mL of anaerobic sewage sludge as inoculant into each of three individual 2 L vessels.
    b. Setup a blank vessel containing only water and inoculant.
    c. Flush each vessel with a non-reactive gas (eg. nitrogen or argon) for 1 minute.
    d. Measure gas volume and concentration in each vessel periodically, as required for a period of 60 days.
4. Calculate the average BMP(t) over time using Equation 3.1.
5. Determine the length of the lag phase and any multi-stage gas production, if present, using the methods described in Sections 5.2.1 and 5.2.2.
6. Fit the results to the first-order decay model shown in Equation 5.1 in order to determine BMP_{ult}.

6.7. Suggestions for Future Research

Future research in the area of BMP analysis has several options. Better understanding of the processes involved, and the various factors that can influence them, is crucial to improving BMP test methods in the future.

This study may be improved upon by verifying the results of the modified BMP method more rigorously. Using a salt solution as the liquid in the water column used for measuring gas volumes would reduce the potential for CO_2 dissolution, and result in greater confidence for gas volume measurements. Periodically analyzing gas samples with a GC to confirm gas composition should add confidence to the measurements obtained by the GEM. Lastly, performing a mass balance analysis based on the initial and final mass of the digested sample, along with the known volume and composition of gas produced would increase confidence in the final results.

To expand upon this study, a close investigation of the chemical and biological environment might provide the ability to better predict and prevent inhibition. This would save time on failed test samples, and possibly lead to greater agreement between replicate sets. Results from this study showed it may also be possible to improve the LOI correlation if more specific ignition temperatures, or temperature ranges, could be found for common plastics. It is likely that ignition at any temperature will also result in the loss of some biodegradable organics, but the correlation would still be significantly improved if the majority of plastics could be accounted for in order that a more precise correlation may be developed.

The aerobic BCP test proposed in this study remains a promising candidate for efficient BMP analysis. There are many unexplored options that may improve the method. Some possibilities are:

- Correlation of partial BCP values to ultimate BMP;
- Improved aeration, or alternatively, water circulation;
- Adding nutrients to improve the health of the biological populations;
- Alternate inoculum sources;
- Testing at elevated temperatures; and,
- Optimizing ratios of inoculum, substrate, and water.

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Appendix A

Calculations for CO_2 Dissolution in Water

0.3 L of Gas in 0.7 L of Water

PV=nRT

P=	94.43	kPa
V=	0.3	L
R=	8.3144621	L kPa/mol K
T=	298	K
n=	0.01143382	mol
$\%CH_4$ =	30	
$\%\ CO_2$ =	70	
n_{CO2} =	0.00800367	

$P_{CO2}=k_Hc$

P_{CO2} =	66.10261	kPa
k_H =	2979.233	L kPa/mol
c =	0.02218779	mol/L
V_w =	0.7	L
max $n_{CO2(aq)}$ =	0.01553146	mol
$\%CO_{2(aq)}$ =	194.054062	

0.9 L of Gas in 0.1 L of Water

PV=nRT

P=	100.32	kPa
V=	0.9	L
R=	8.3144621	L kPa/mol K
T=	298	K
n=	0.0364397	mol
$\%CH_4$ =	30	
$\%\ CO_2$ =	70	
n_{CO2} =	0.02550779	

$P_{CO2}=k_Hc$

P_{CO2} =	70.22323	kPa
k_H =	2979.233	L kPa/mol
c =	0.02357091	mol/L
V_w =	0.1	L
$n_{CO2(aq)}$ =	0.00235709	mol
$\%CO_{2(aq)}$ =	9.24066961	

Input Value
Constant
Caluclated Value

Assuming a 1 L column, 1 m high
Atmospheric Pressure = 101.3 kPa
Unit Weight of Water = 9.811 kPa/m

0.5 L of Gas in 0.5 L of Water

PV=nRT

P=	94.43	kPa
V=	0.5	L
R=	8.3144621	L kPa/mol K
T=	298	K
n=	0.01905637	mol
$\%CH_4$ =	30	
$\%CO_2$ =	70	
n_{CO2} =	0.01333946	

$P_{CO2}=k_Hc$

P_{CO2} =	66.10261	kPa
k_H =	2979.233	L kPa/mol
c =	0.02218779	mol/L
V_w =	0.5	L
max $n_{CO2(aq)}$ =	0.0110939	mol
$\%CO_{2(aq)}$ =	83.1660265	

0.6 L of Gas in 0.4 L of Water

PV=nRT

P=	100.32	kPa
V=	0.6	L
R=	8.3144621	L kPa/mol K
T=	298	K
n=	0.02429314	mol
$\%CH_4$ =	30	
$\%CO_2$ =	70	
n_{CO2} =	0.0170052	

$P_{CO2}=k_Hc$

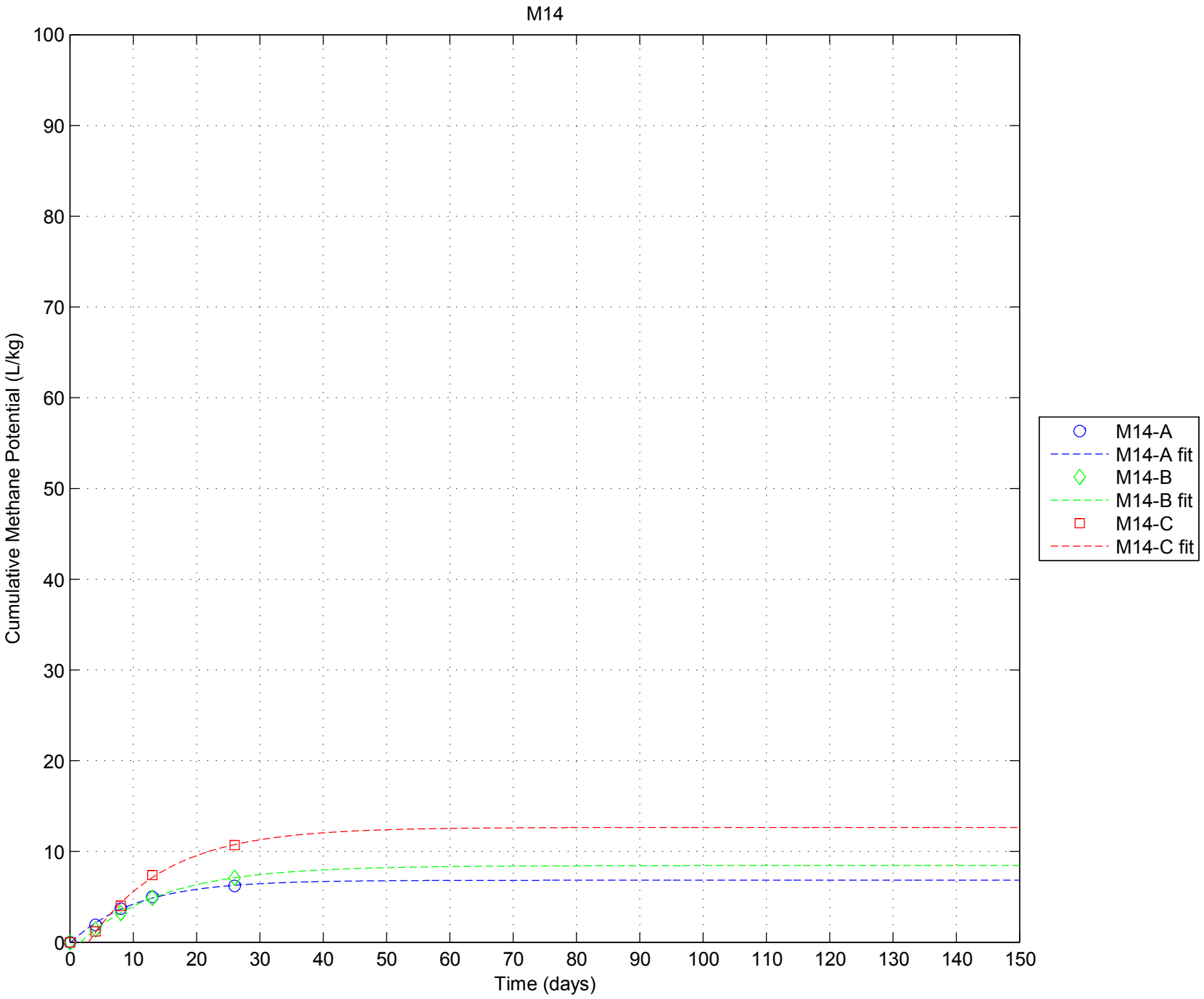
P_{CO2} =	70.22323	kPa
k_H =	2979.233	L kPa/mol
c =	0.02357091	mol/L
V_w =	0.4	L
$n_{CO2(aq)}$ =	0.00942836	mol
$\%CO_{2(aq)}$ =	55.4440177	

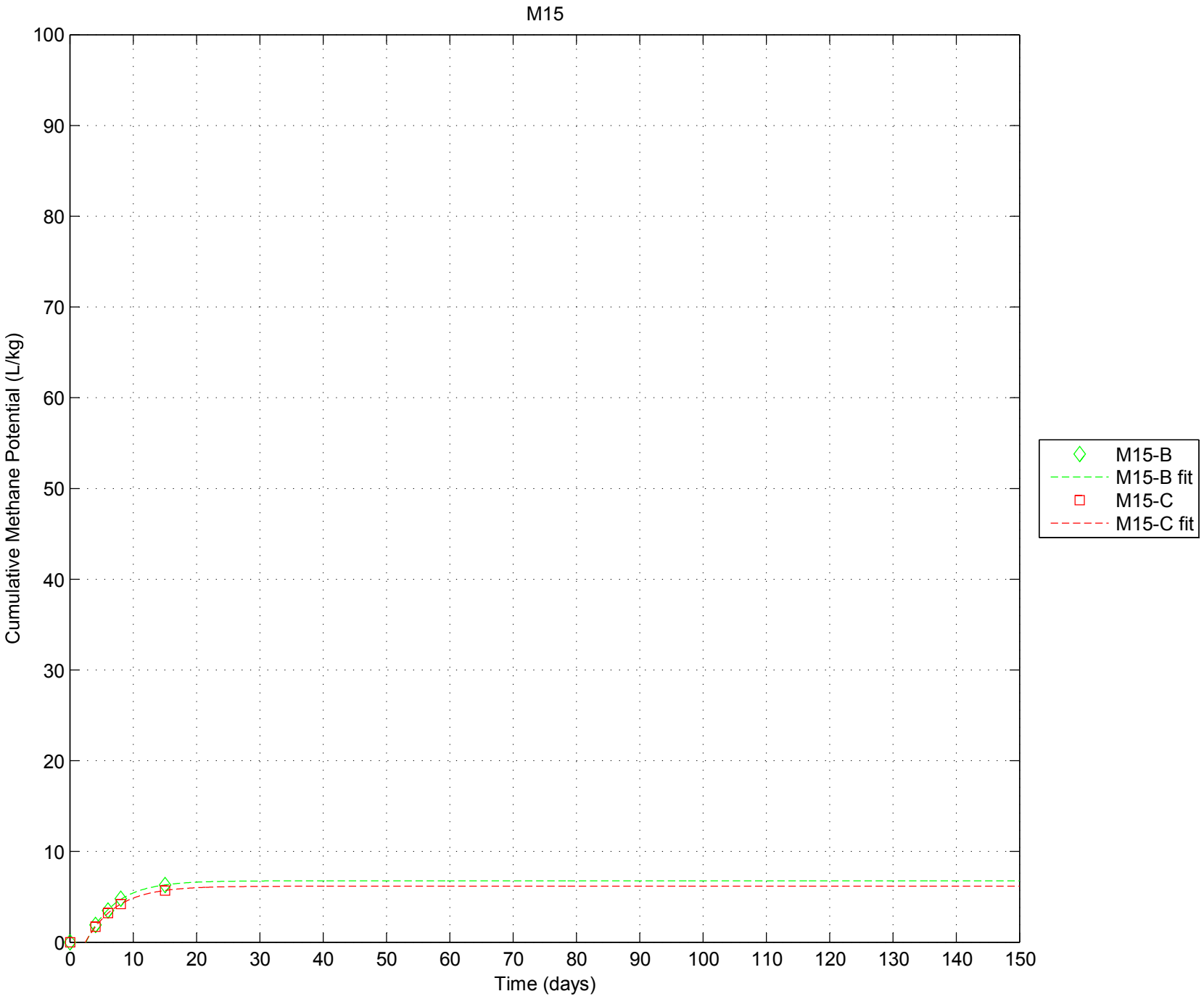
Input Value
Constant
Caluclated Value

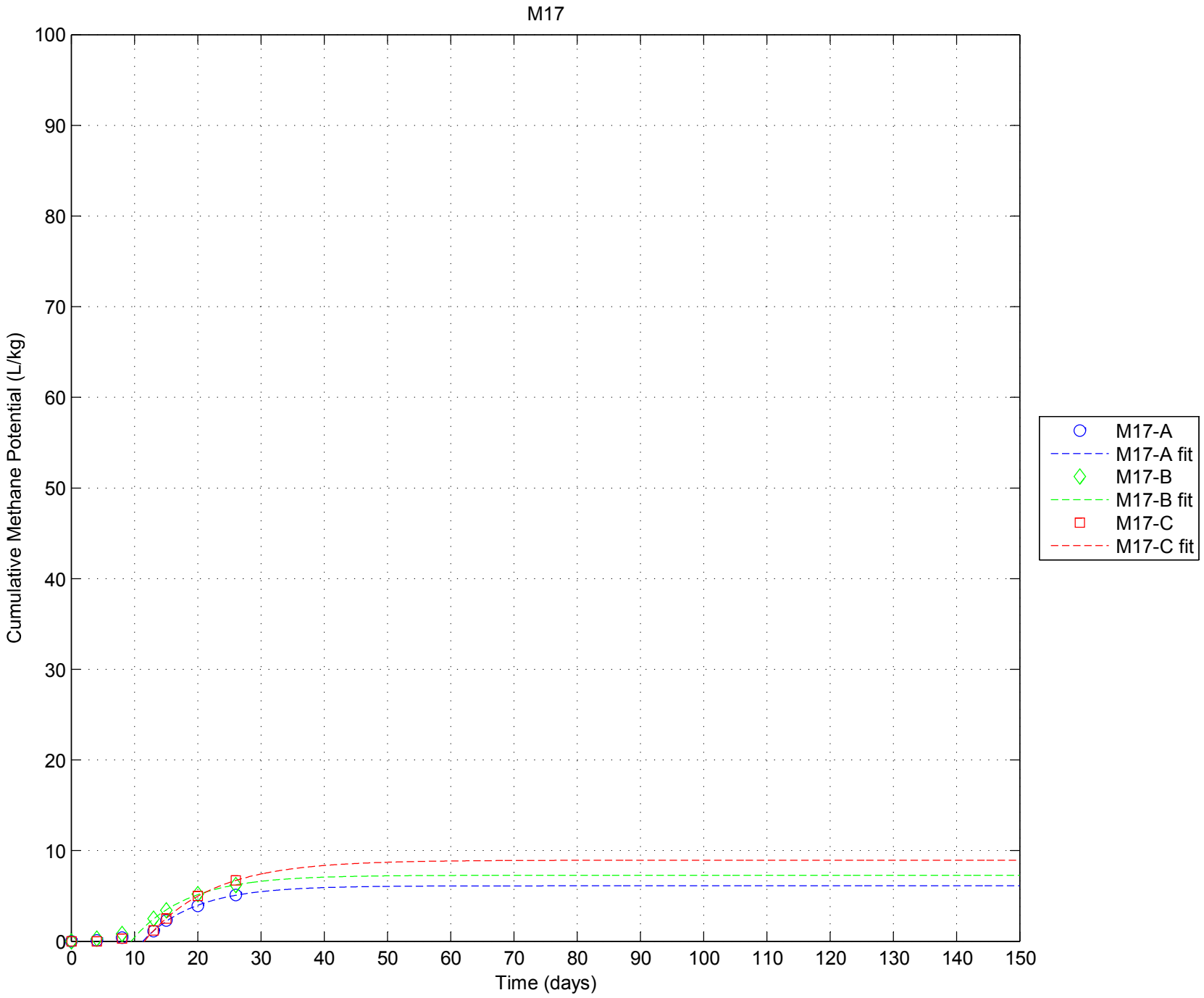
Assuming a 1 L column, 1 m high
Atmospheric Pressure = 101.3 kPa
Unit Weight of Water = 9.811 kPa/m

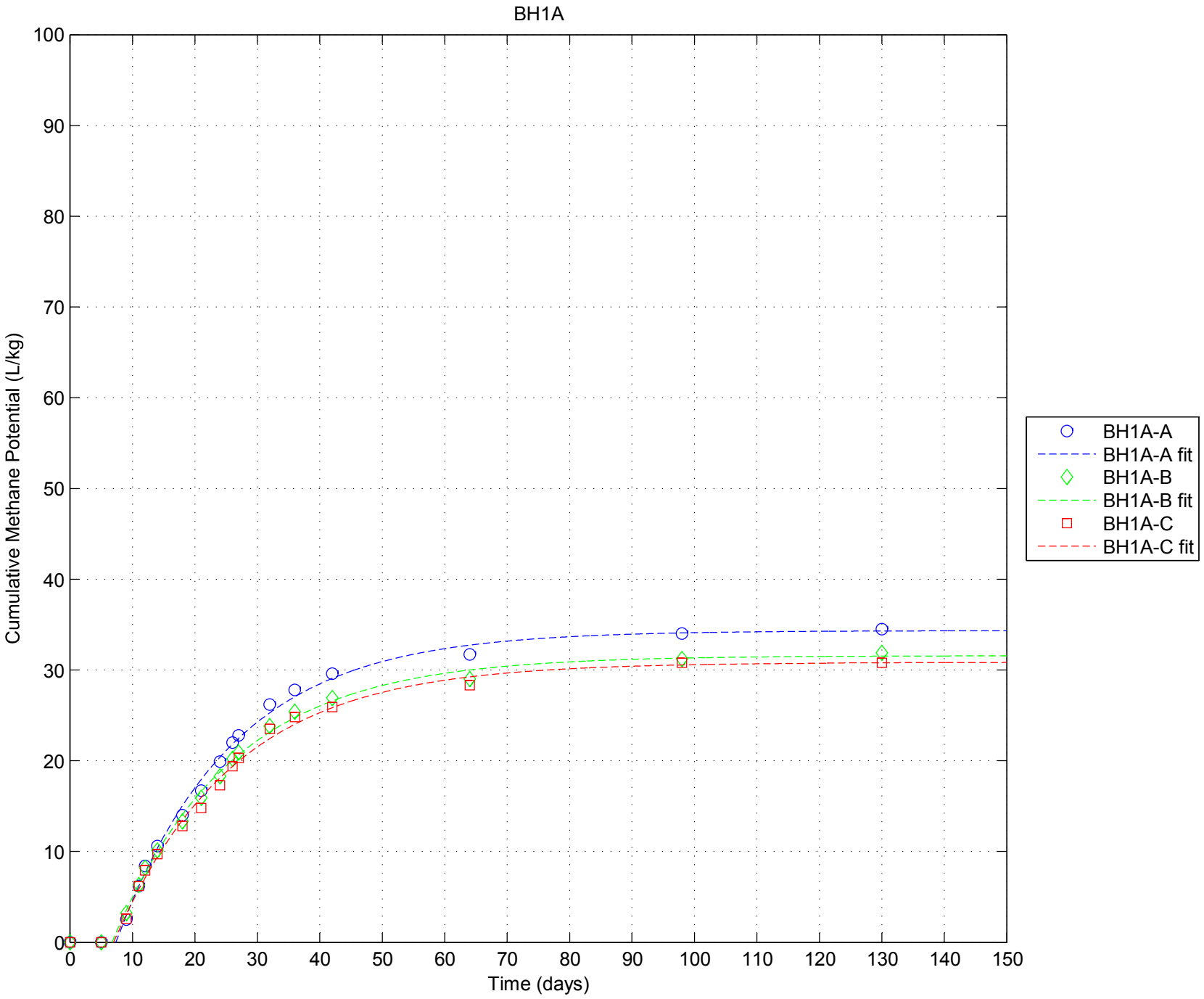
Appendix B

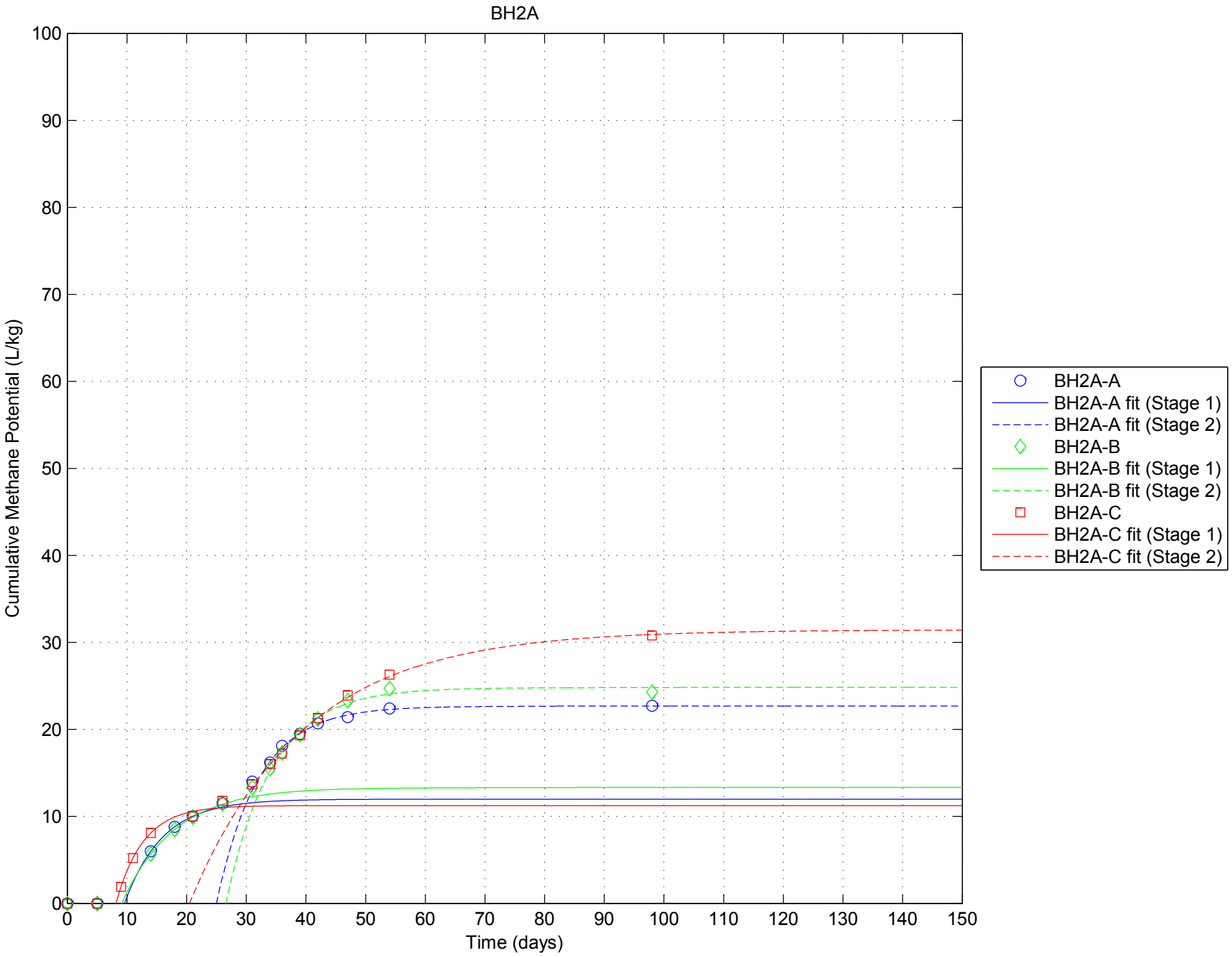
BMP Plots

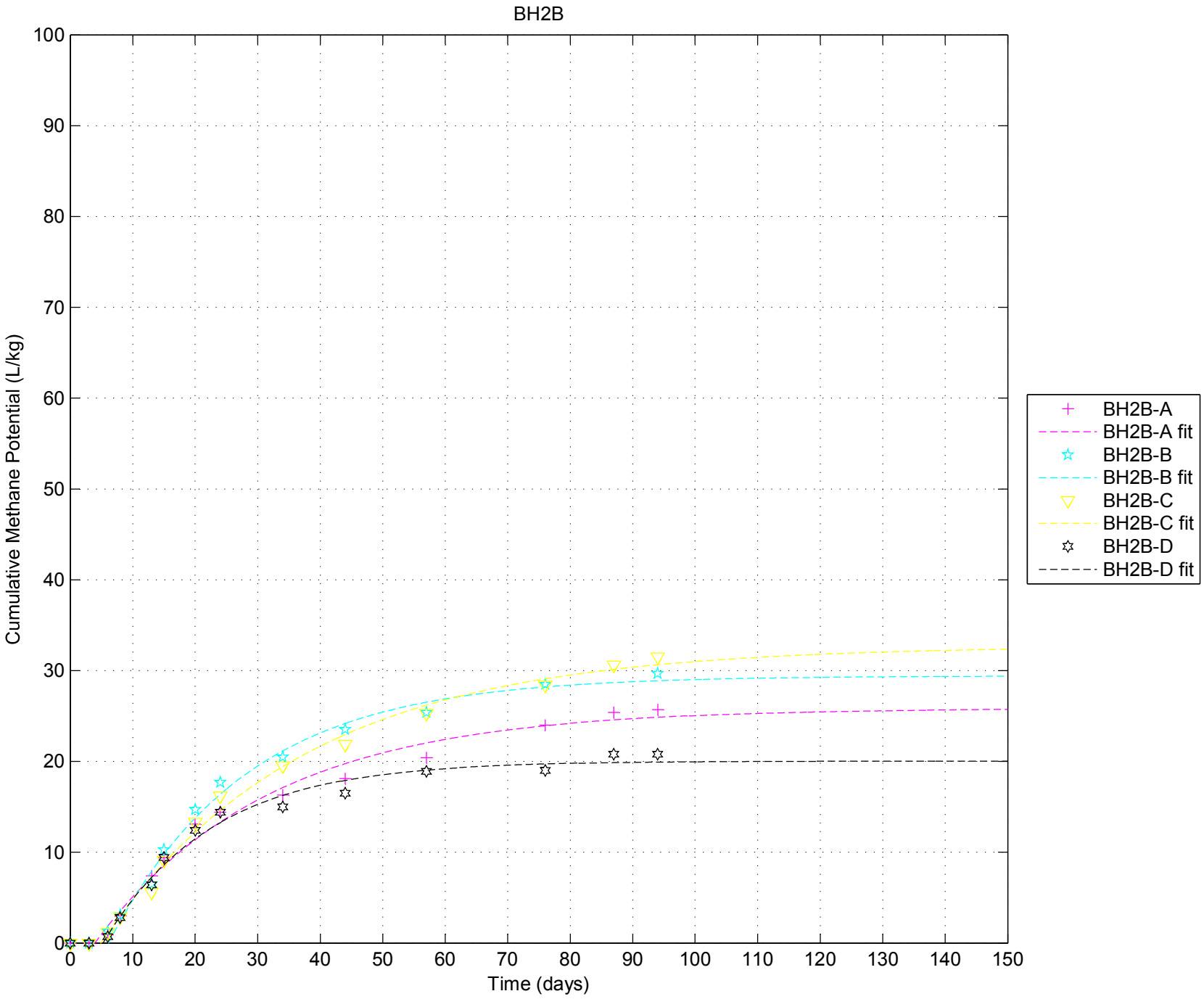

M14
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
M14-A
M14-A fit
M14-B
M14-B fit
M14-C
M14-C fit

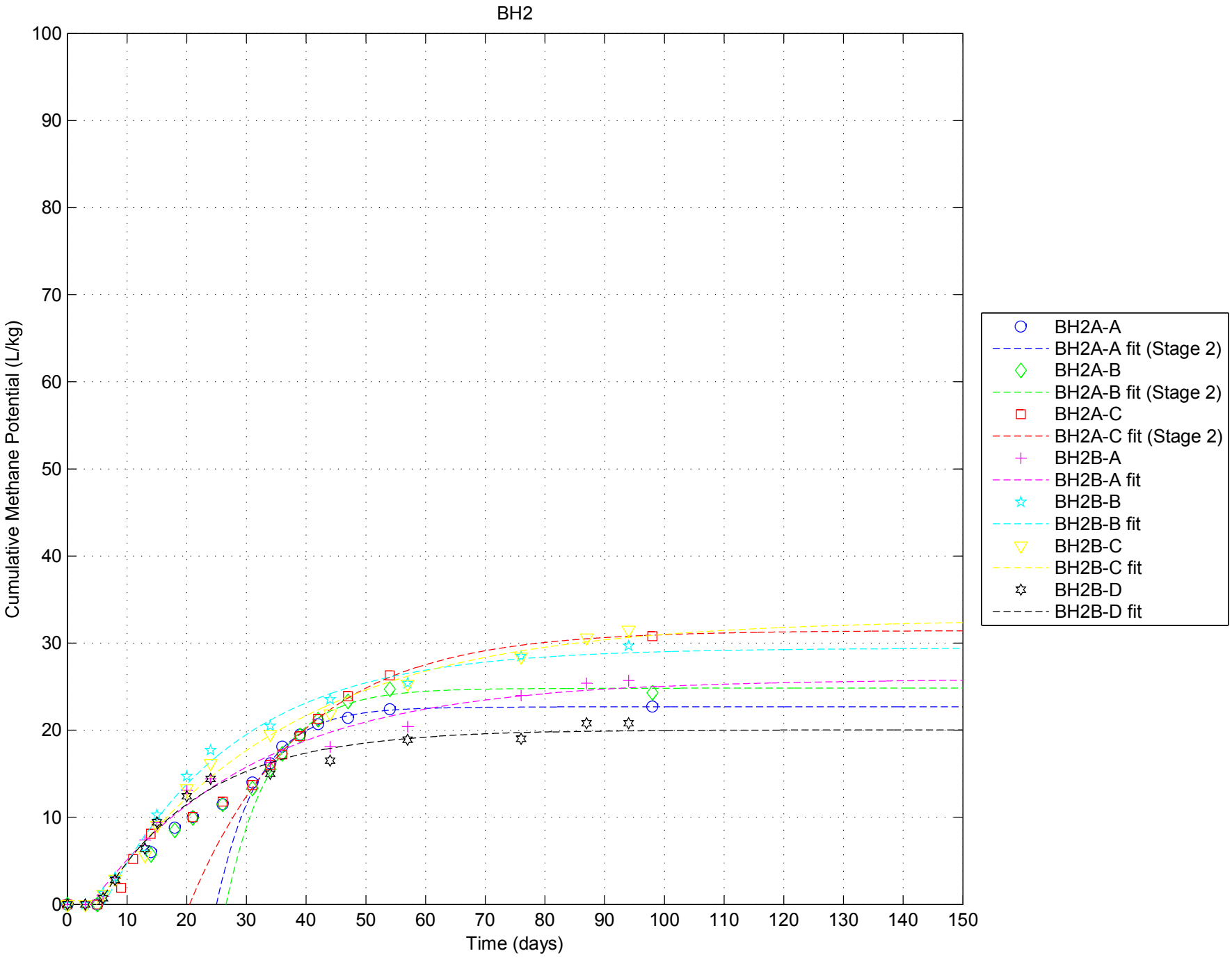

M15
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
M15-B
M15-B fit
M15-C
M15-C fit

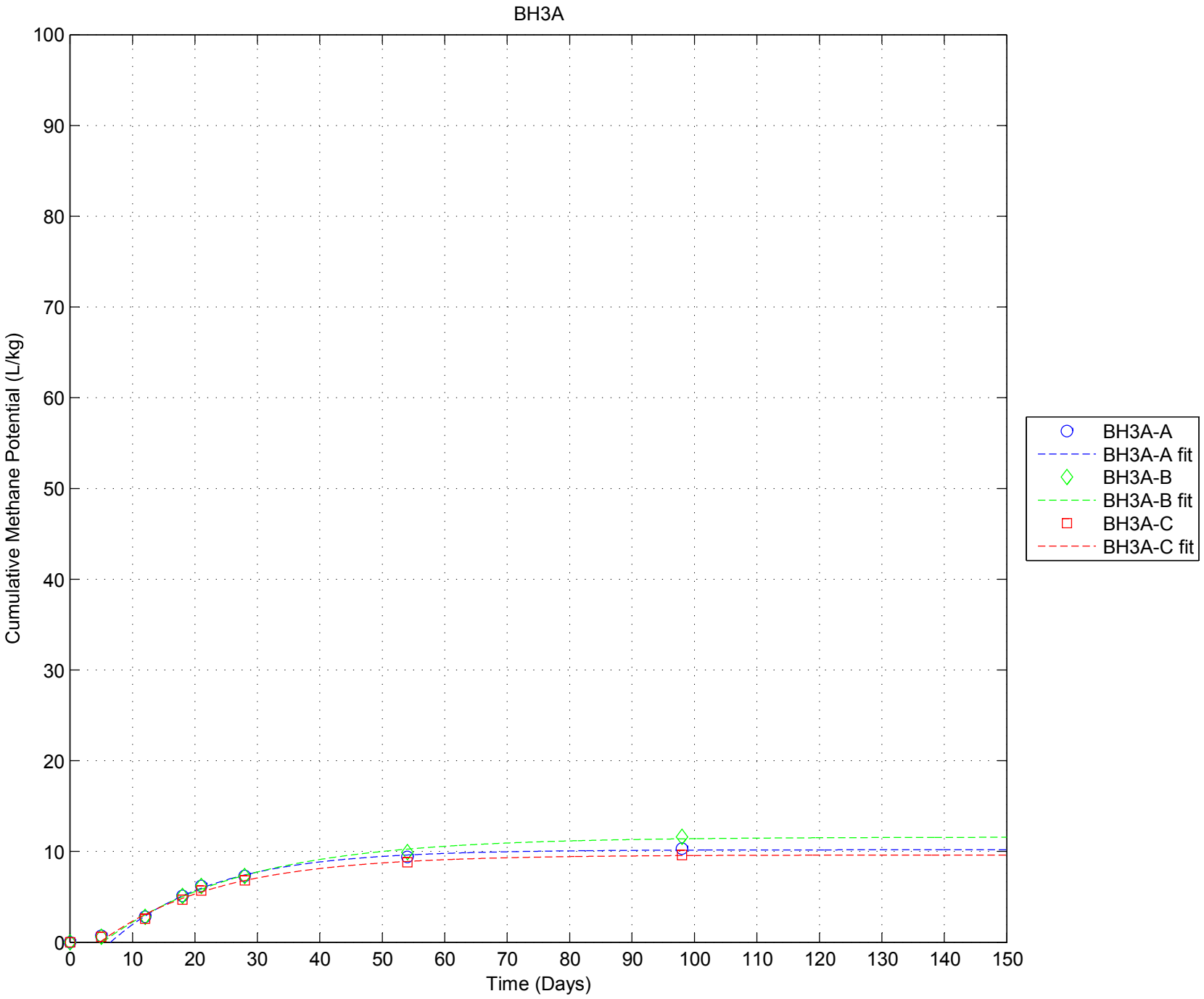

M17
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
M17-A
M17-A fit
M17-B
M17-B fit
M17-C
M17-C fit

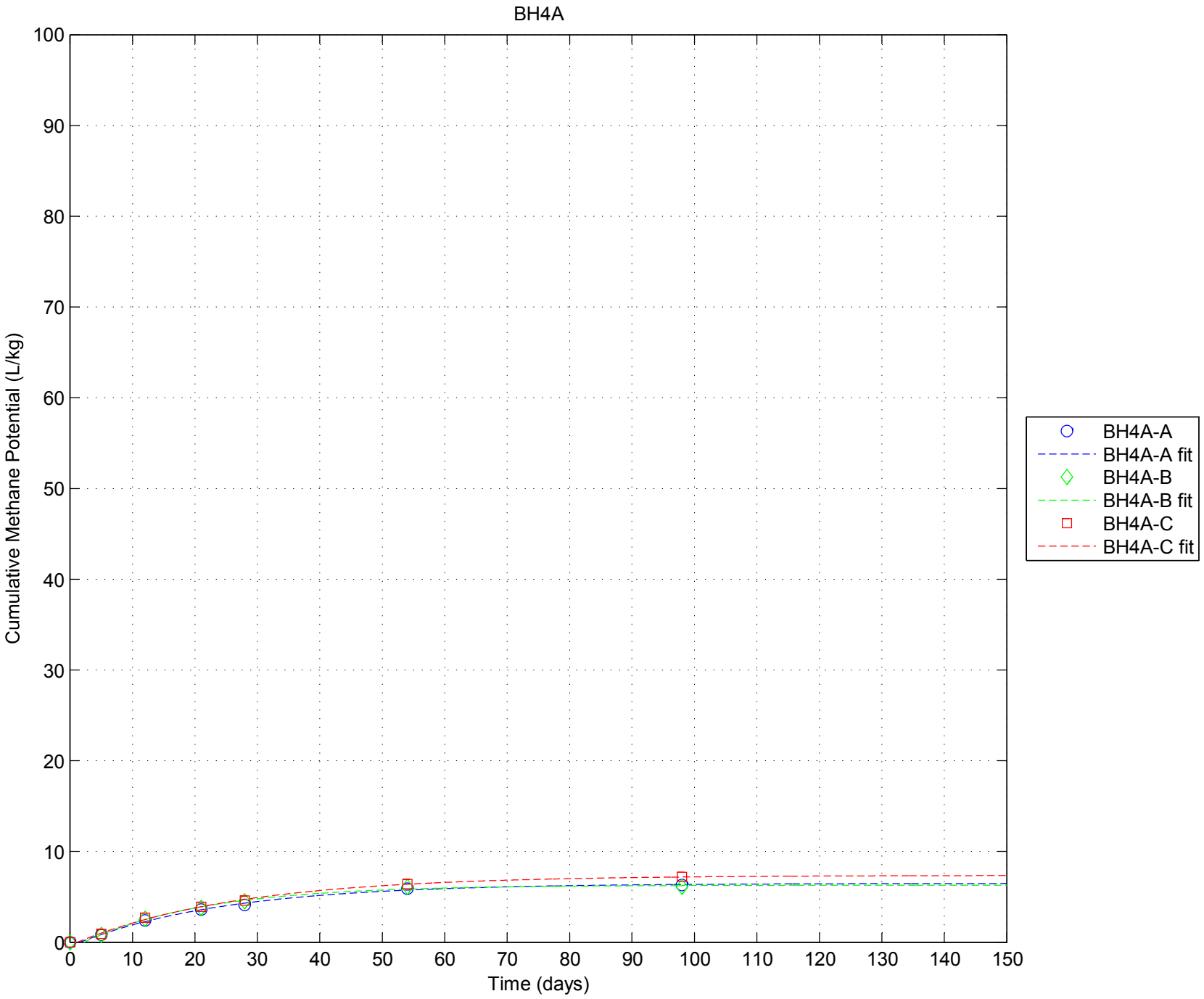

BH1A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH1A-A
BH1A-A fit
BH1A-B
BH1A-B fit
BH1A-C
BH1A-C fit

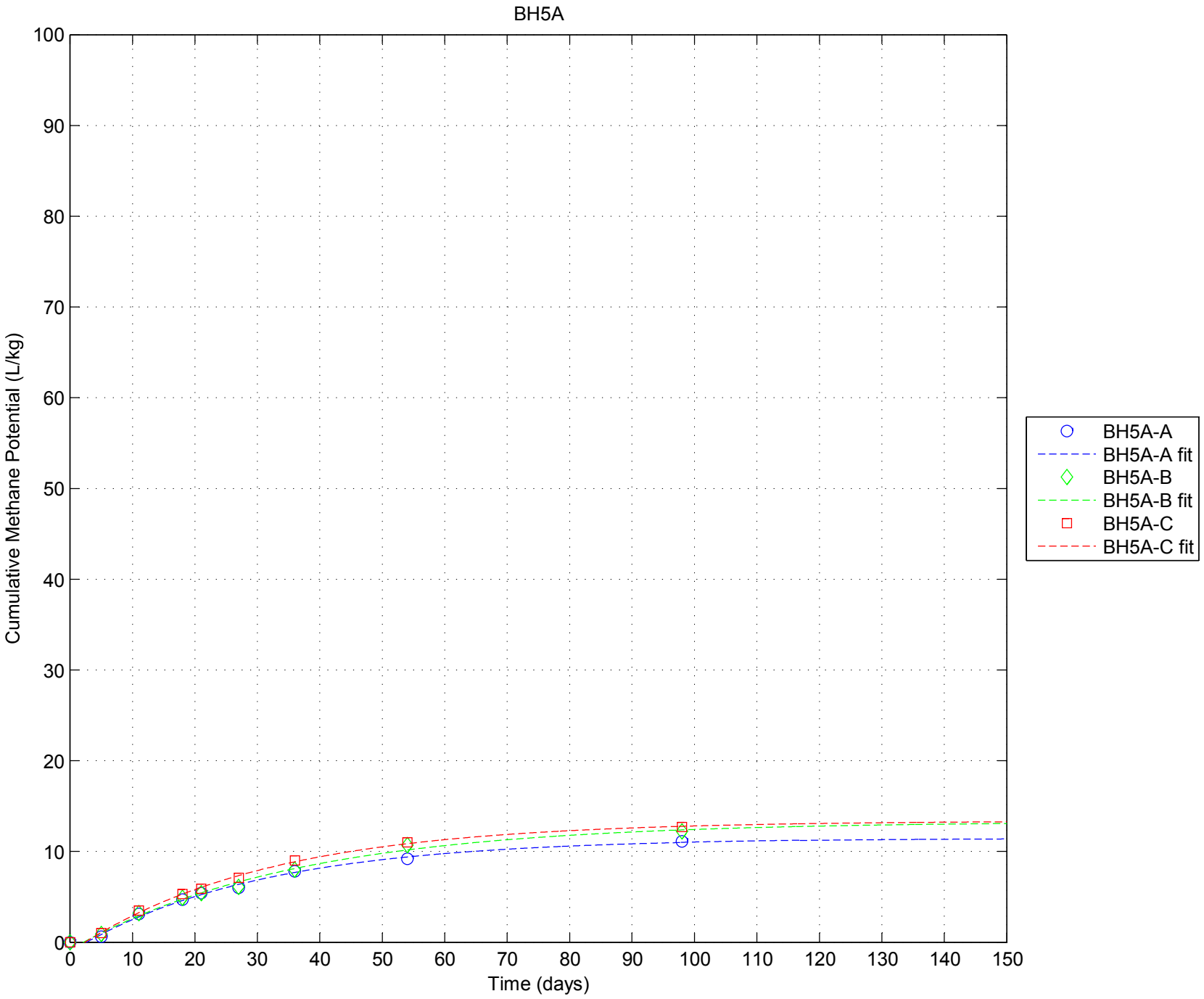

BH2A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH2A-A
BH2A-A fit (Stage 1)
BH2A-A fit (Stage 2)
BH2A-B
BH2A-B fit (Stage 1)
BH2A-B fit (Stage 2)
BH2A-C
BH2A-C fit (Stage 1)
BH2A-C fit (Stage 2)

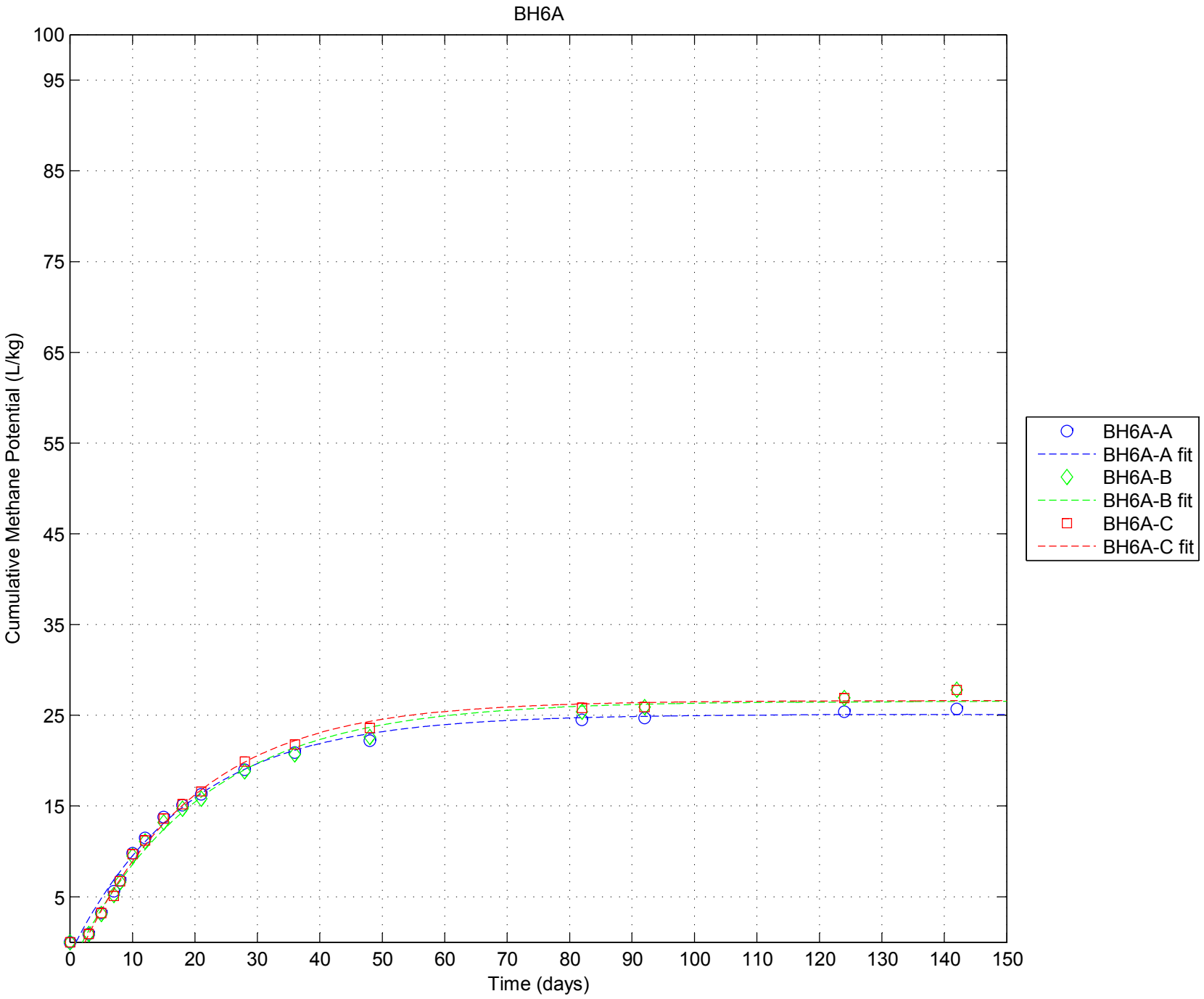

BH2B
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH2B-A
BH2B-A fit
BH2B-B
BH2B-B fit
BH2B-C
BH2B-C fit
BH2B-D
BH2B-D fit

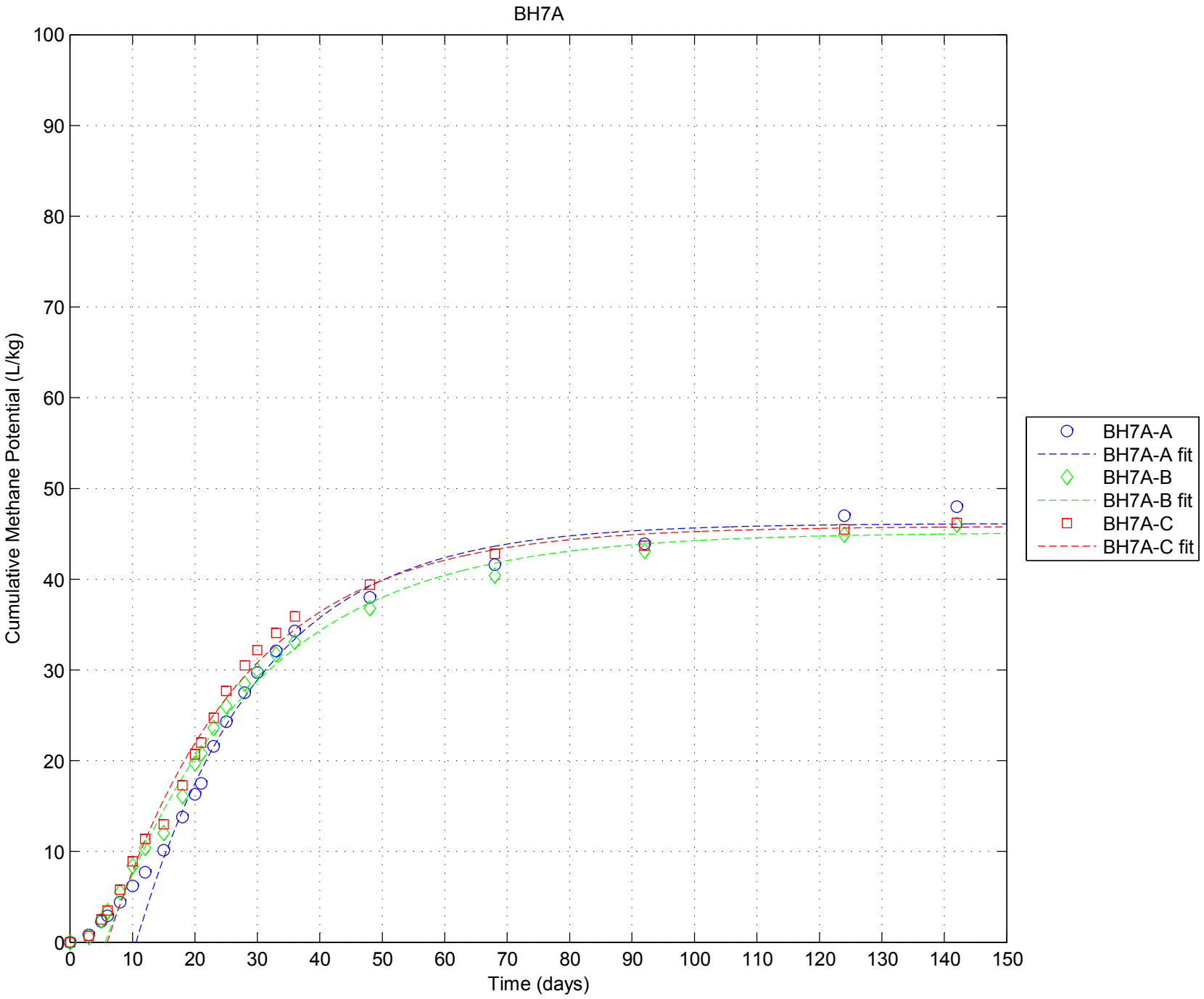

BH2
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH2A-A
BH2A-A fit (Stage 2)
BH2A-B
BH2A-B fit (Stage 2)
BH2A-C
BH2A-C fit (Stage 2)
BH2B-A
BH2B-A fit
BH2B-B
BH2B-B fit
BH2B-C
BH2B-C fit
BH2B-D
BH2B-D fit

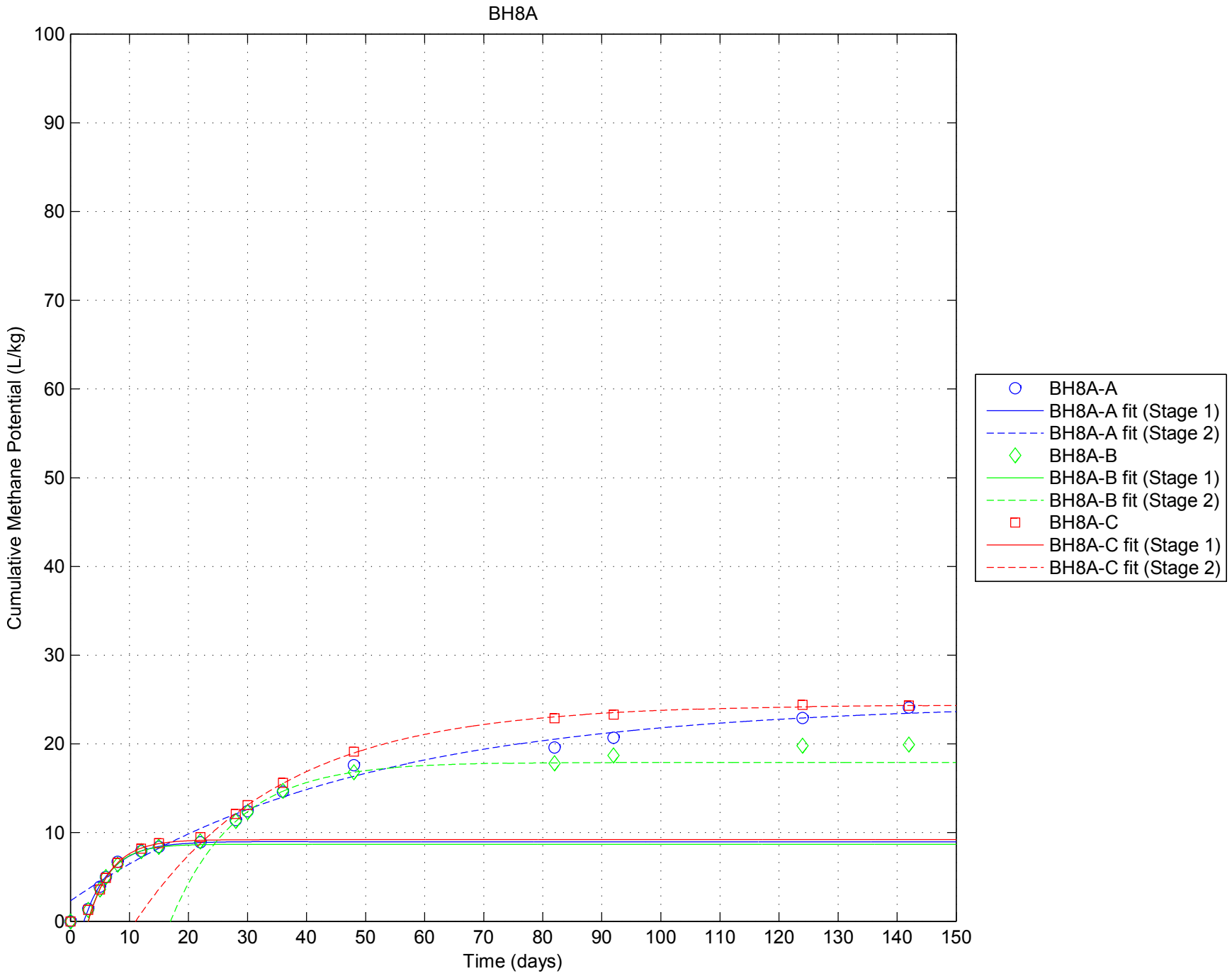

BH3A
Cumulative Methane Potential (L/kg)
Time (Days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH3A-A
BH3A-A fit
BH3A-B
BH3A-B fit
BH3A-C
BH3A-C fit

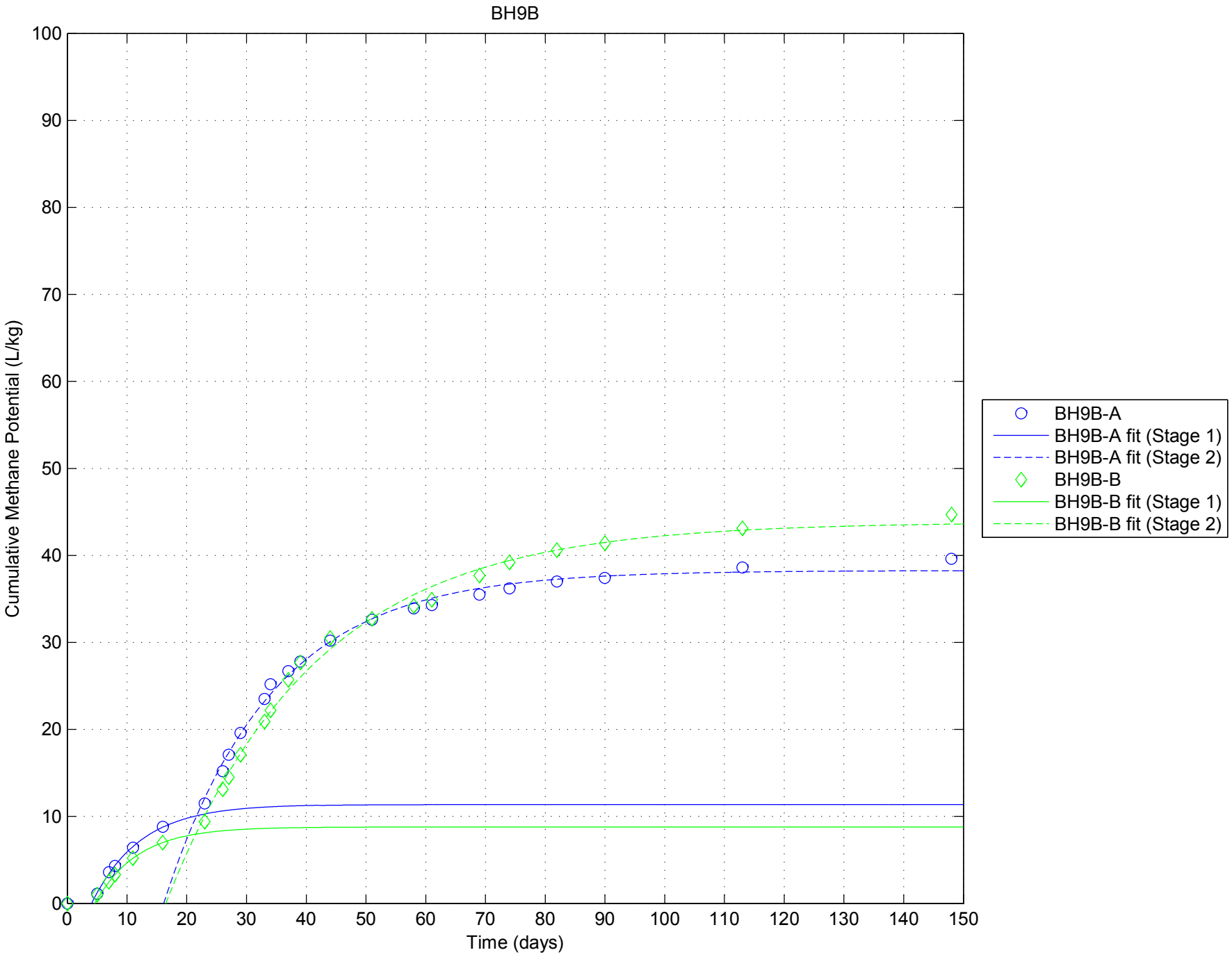

BH4A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH4A-A
BH4A-A fit
BH4A-B
BH4A-B fit
BH4A-C
BH4A-C fit

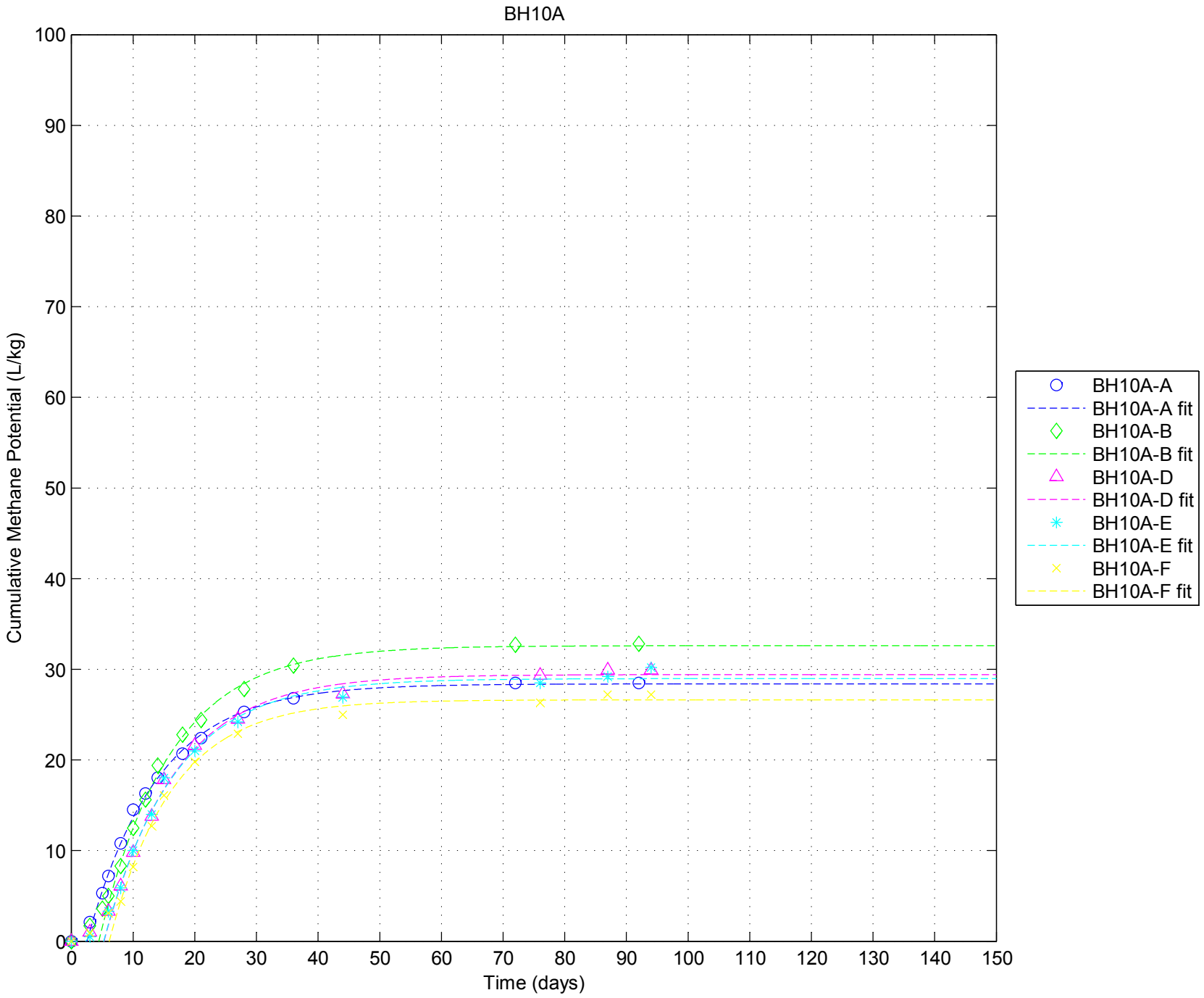

BH5A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH5A-A
BH5A-A fit
BH5A-B
BH5A-B fit
BH5A-C
BH5A-C fit

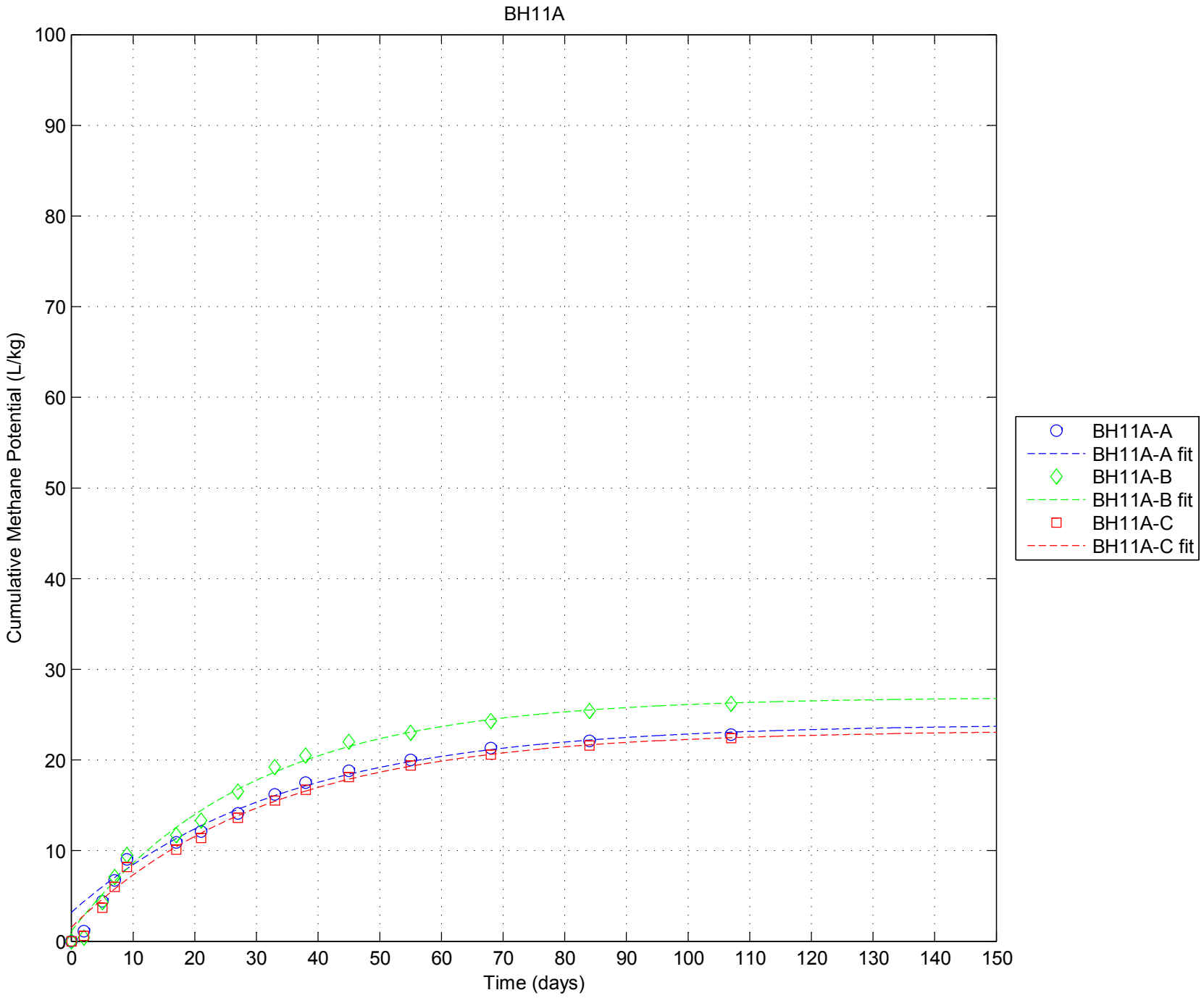

BH6A
Cumulative Methane Potential (L/kg)
Time (days)
100
95
85
75
65
55
45
35
25
15
5
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH6A-A
BH6A-A fit
BH6A-B
BH6A-B fit
BH6A-C
BH6A-C fit

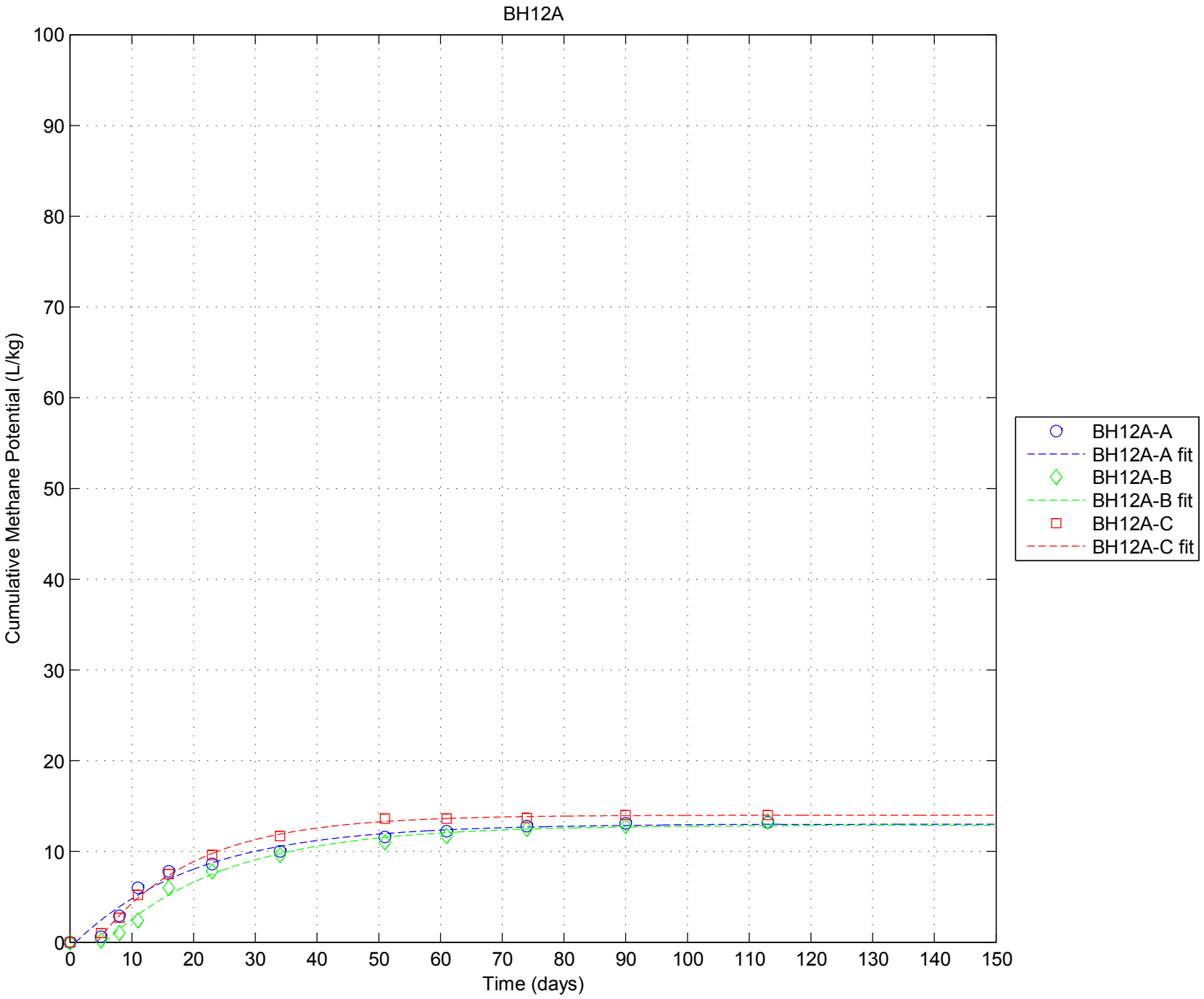

BH7A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH7A-A
BH7A-A fit
BH7A-B
BH7A-B fit
BH7A-C
BH7A-C fit

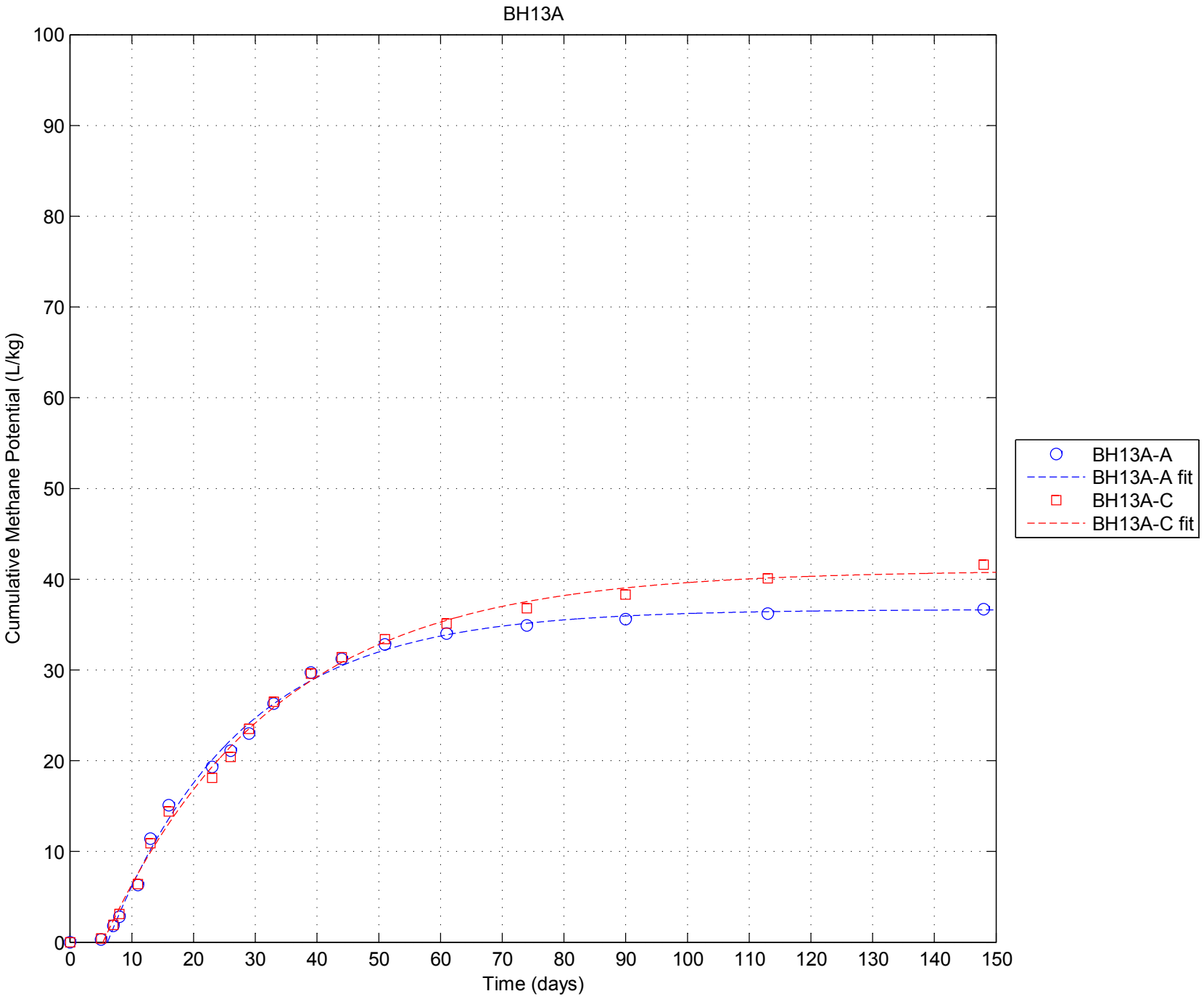

BH8A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH8A-A
BH8A-A fit (Stage 1)
BH8A-A fit (Stage 2)
BH8A-B
BH8A-B fit (Stage 1)
BH8A-B fit (Stage 2)
BH8A-C
BH8A-C fit (Stage 1)
BH8A-C fit (Stage 2)

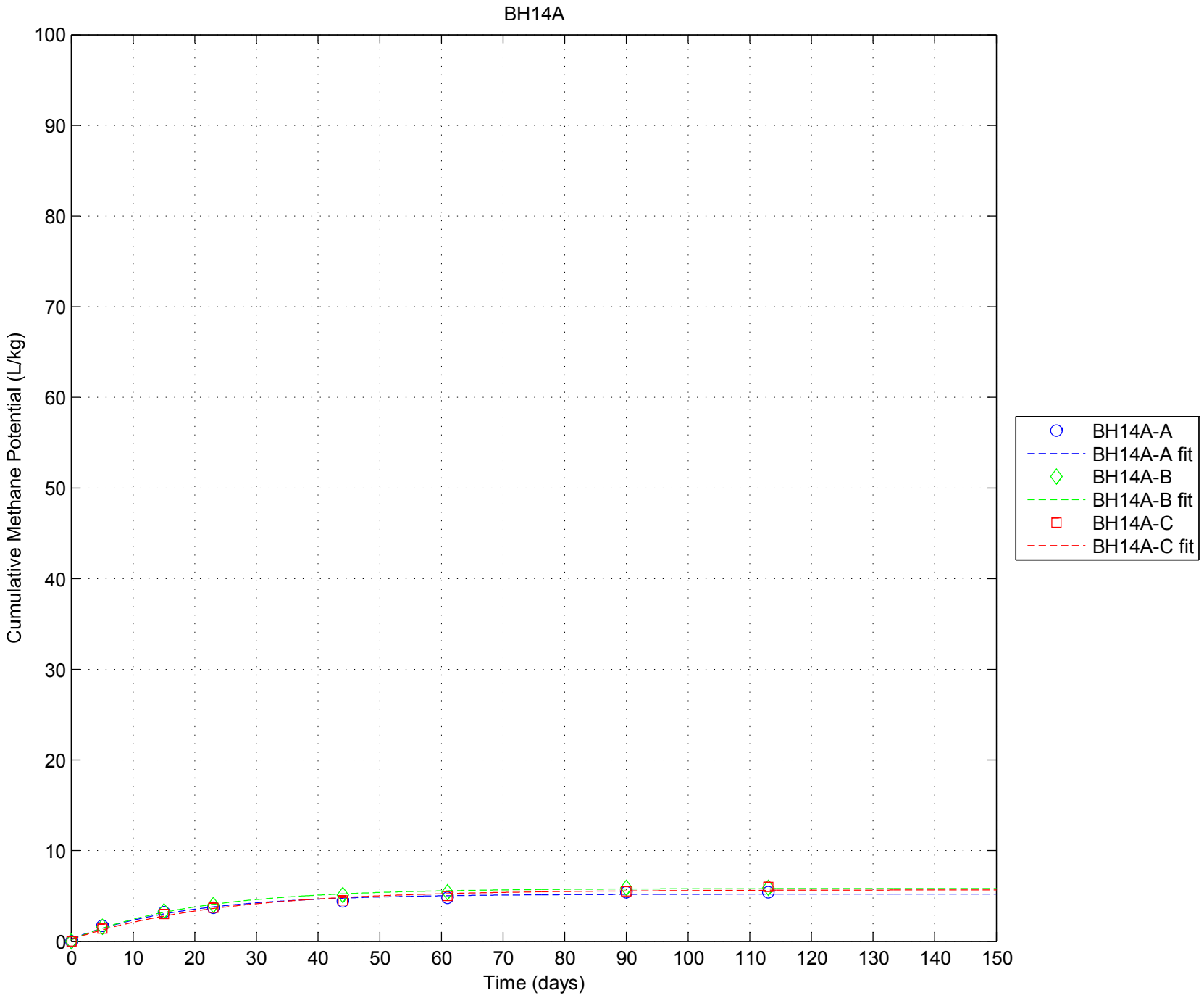

BH9B
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH9B-A
BH9B-A fit (Stage 1)
BH9B-A fit (Stage 2)
BH9B-B
BH9B-B fit (Stage 1)
BH9B-B fit (Stage 2)

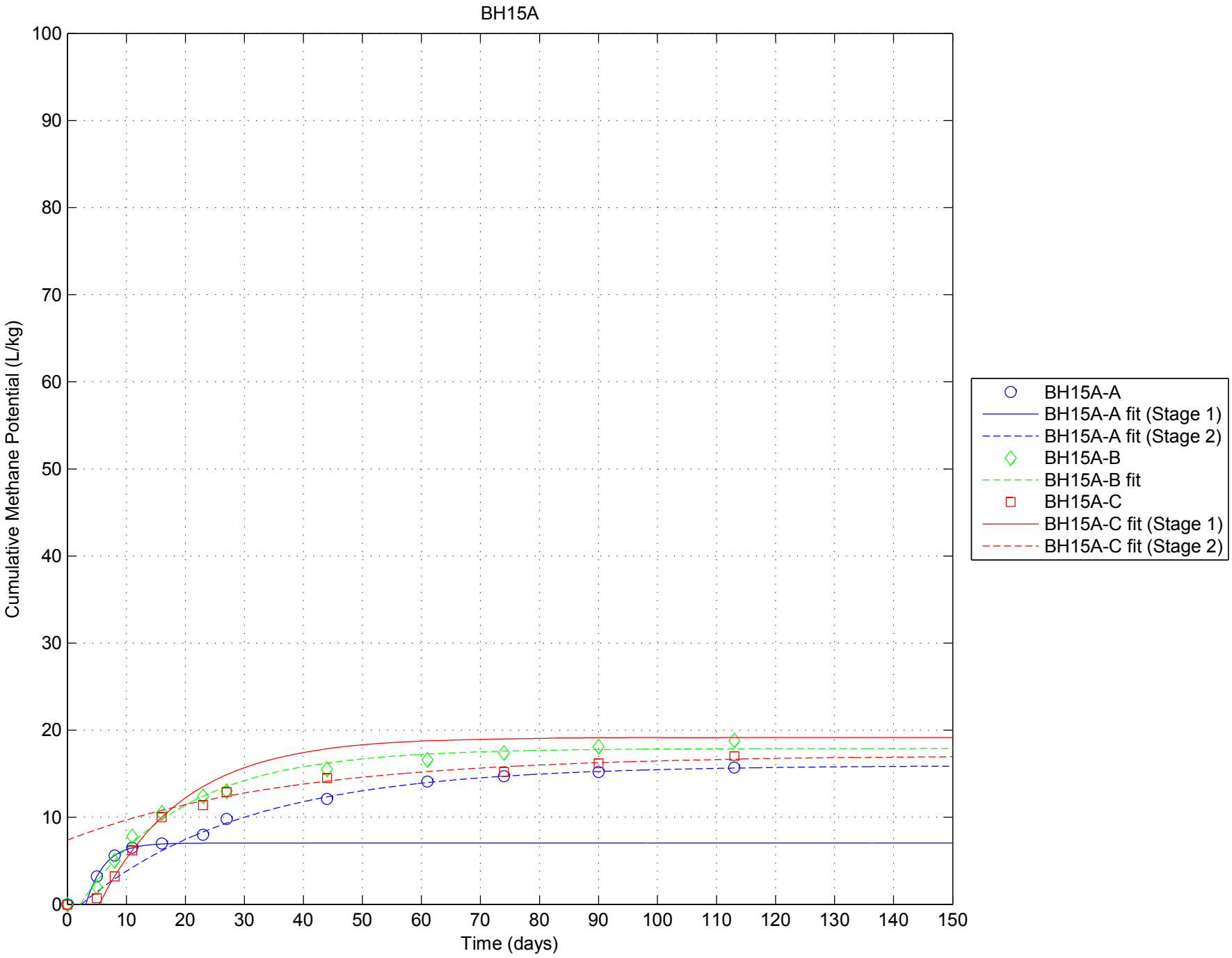

BH10A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH10A-A
BH10A-A fit
BH10A-B
BH10A-B fit
BH10A-D
BH10A-D fit
BH10A-E
BH10A-E fit
BH10A-F
BH10A-F fit

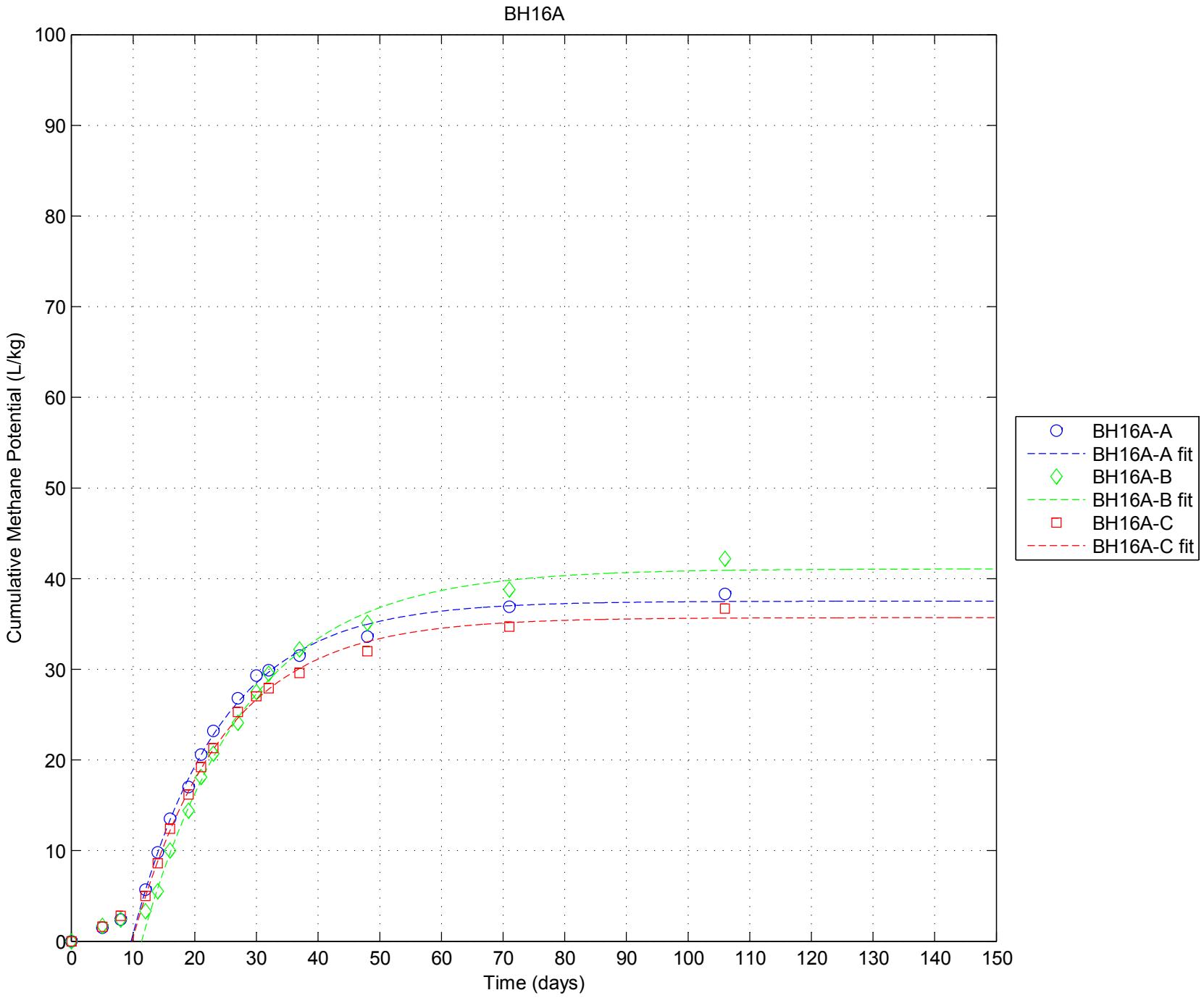

BH11A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH11A-A
BH11A-A fit
BH11A-B
BH11A-B fit
BH11A-C
BH11A-C fit

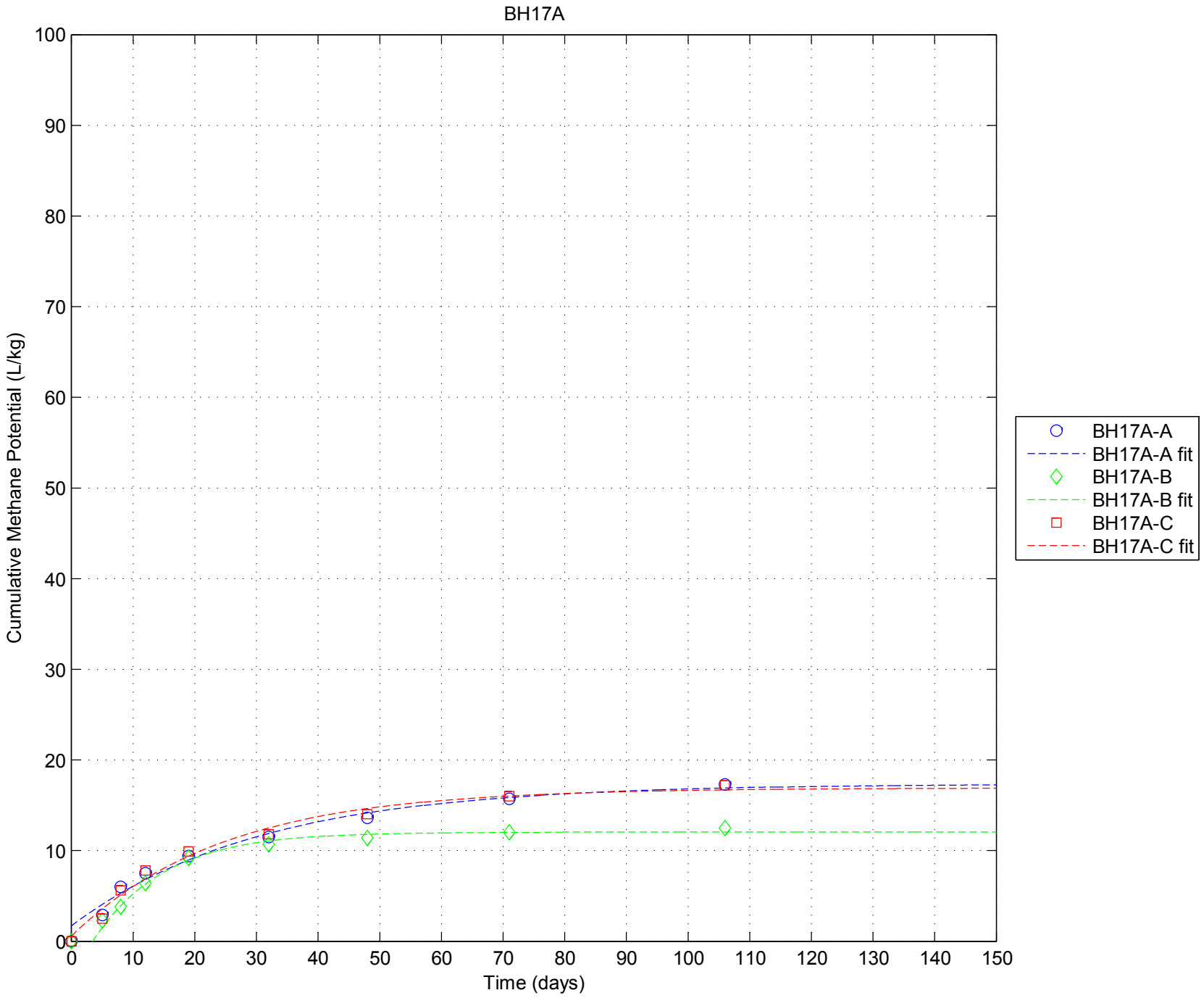

BH12A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH12A-A
BH12A-A fit
BH12A-B
BH12A-B fit
BH12A-C
BH12A-C fit

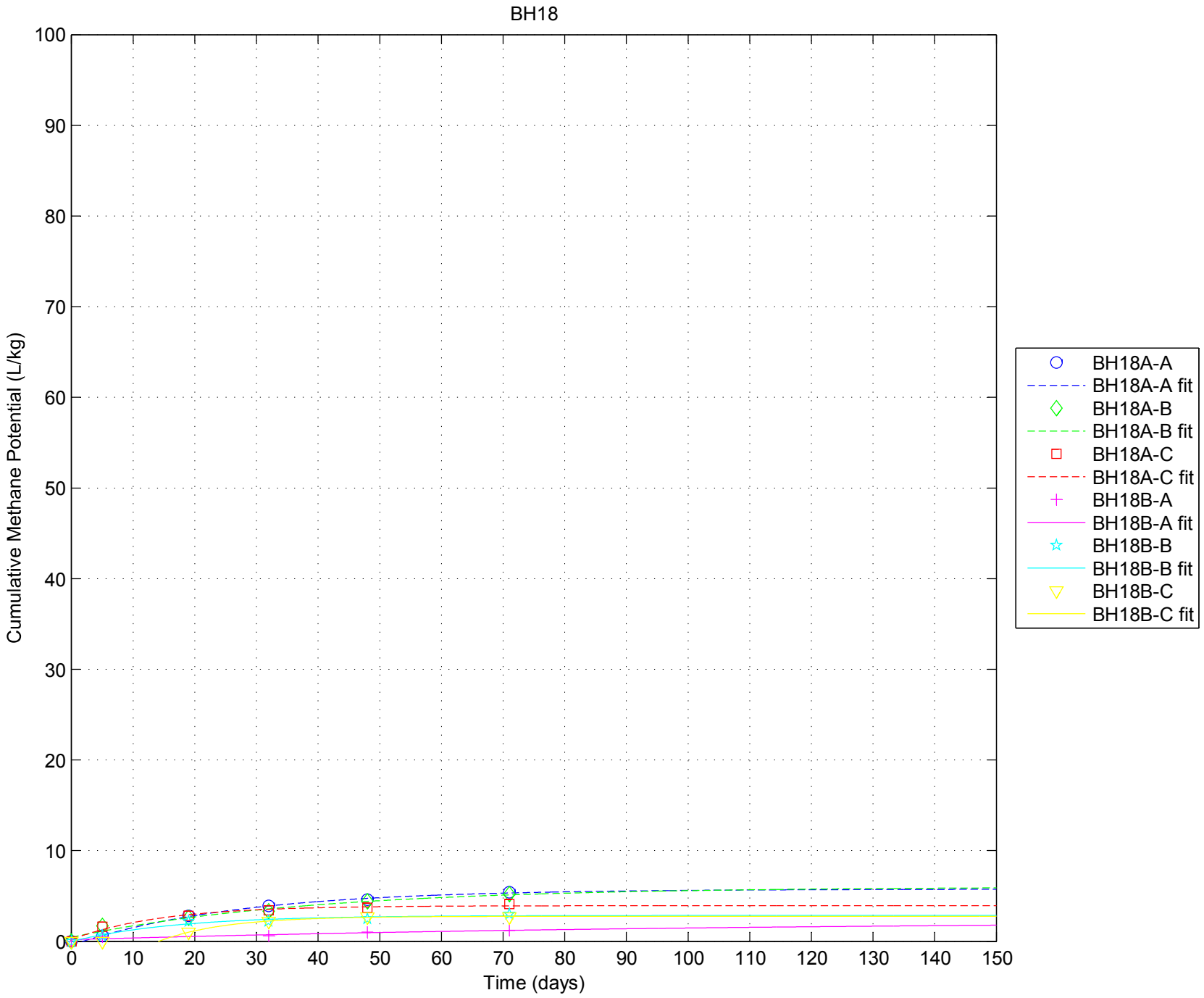

BH13A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH13A-A
BH13A-A fit
BH13A-C
BH13A-C fit

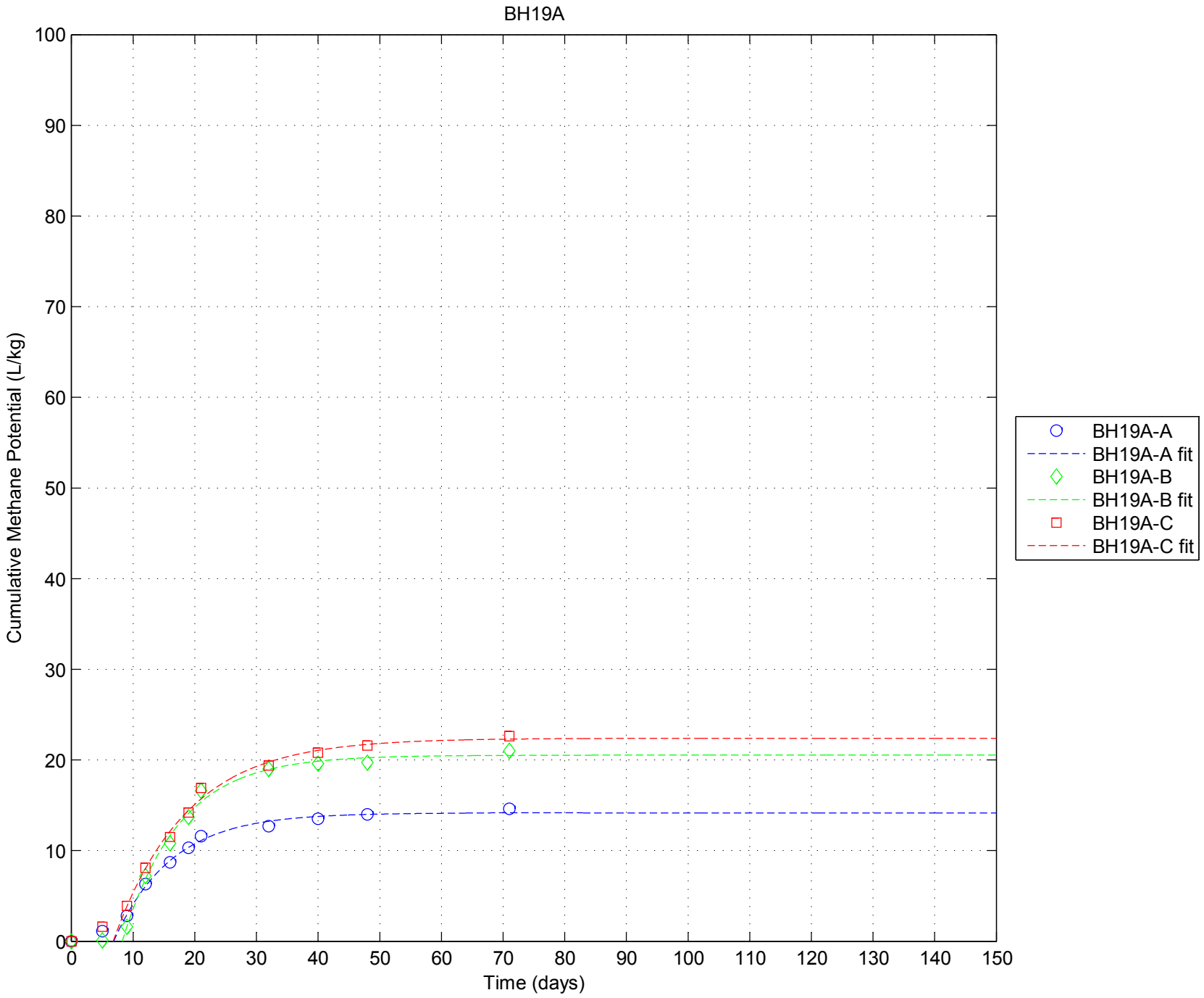

BH14A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH14A-A
BH14A-A fit
BH14A-B
BH14A-B fit
BH14A-C
BH14A-C fit

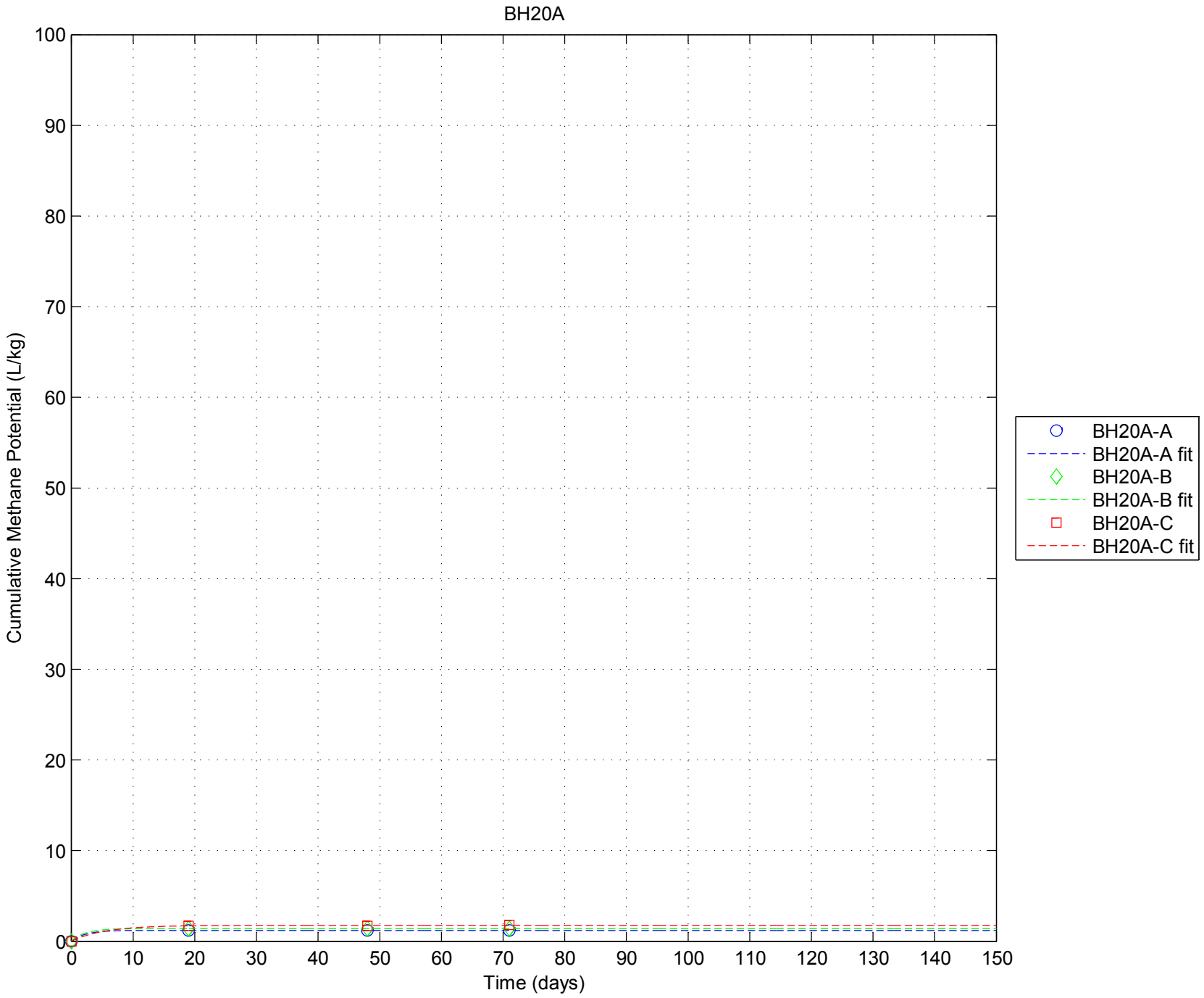

BH15A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH15A-A
BH15A-A fit (Stage 1)
BH15A-A fit (Stage 2)
BH15A-B
BH15A-B fit
BH15A-C
BH15A-C fit (Stage 1)
BH15A-C fit (Stage 2)

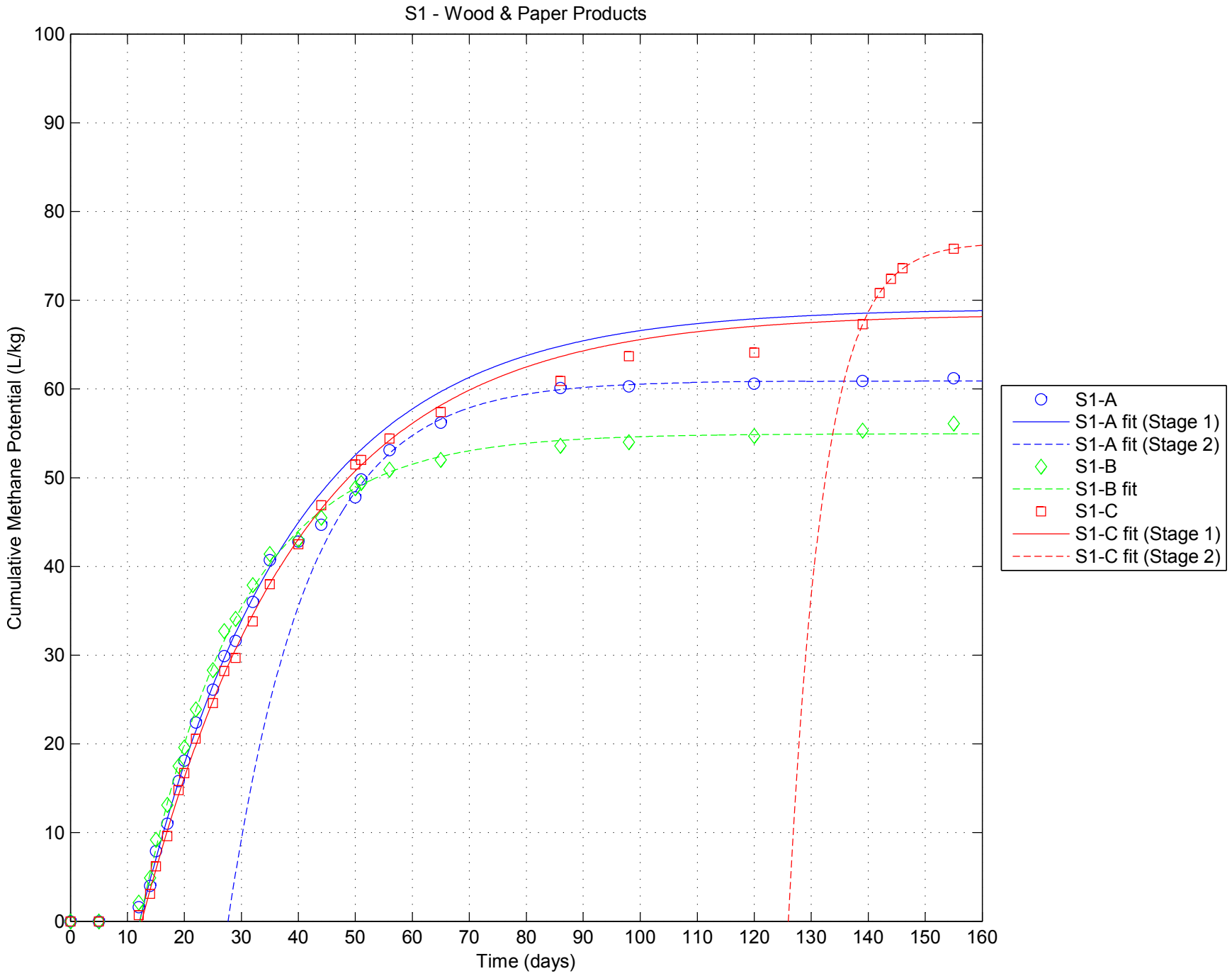

BH16A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH16A-A
BH16A-A fit
BH16A-B
BH16A-B fit
BH16A-C
BH16A-C fit

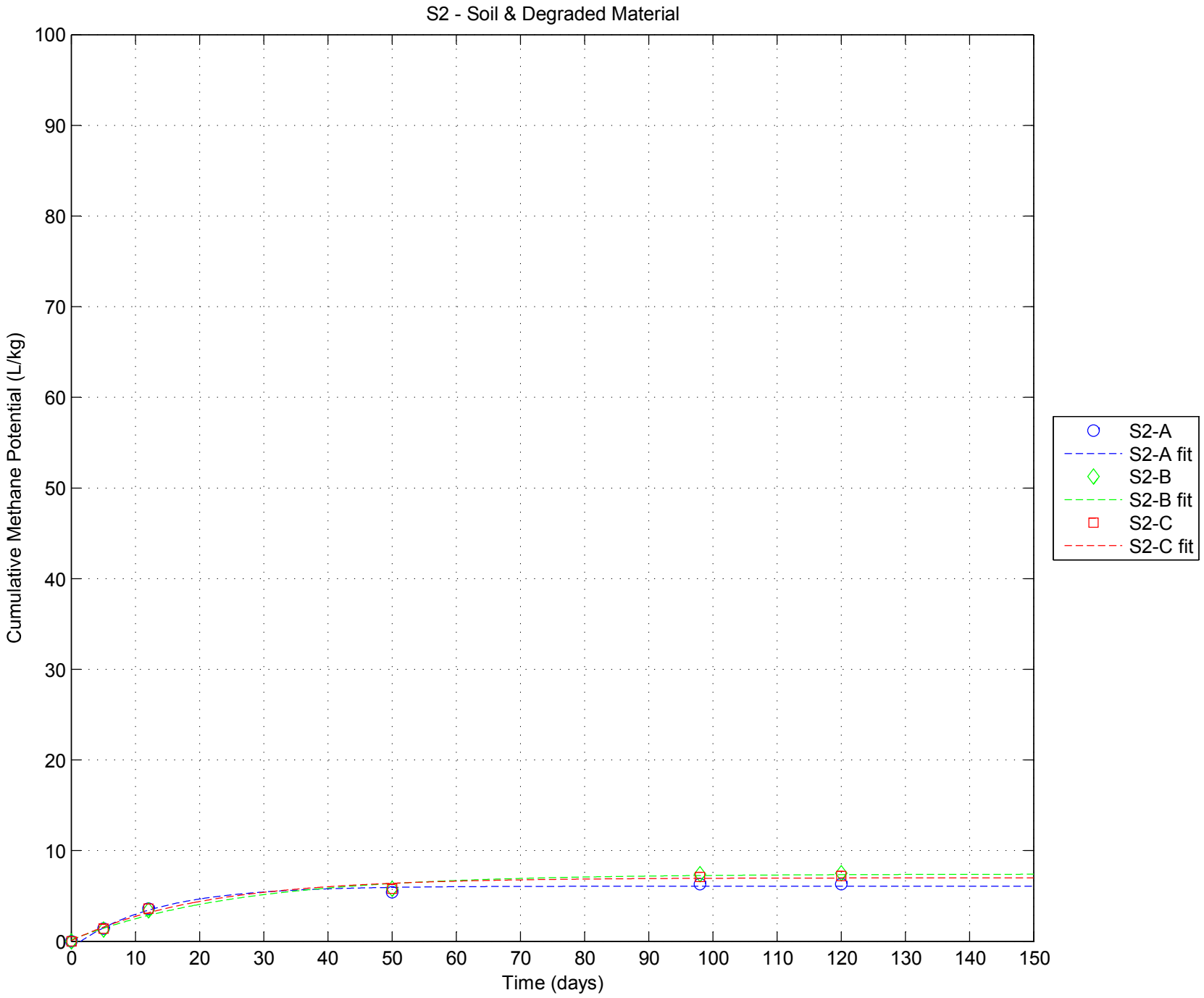

BH17A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH17A-A
BH17A-A fit
BH17A-B
BH17A-B fit
BH17A-C
BH17A-C fit

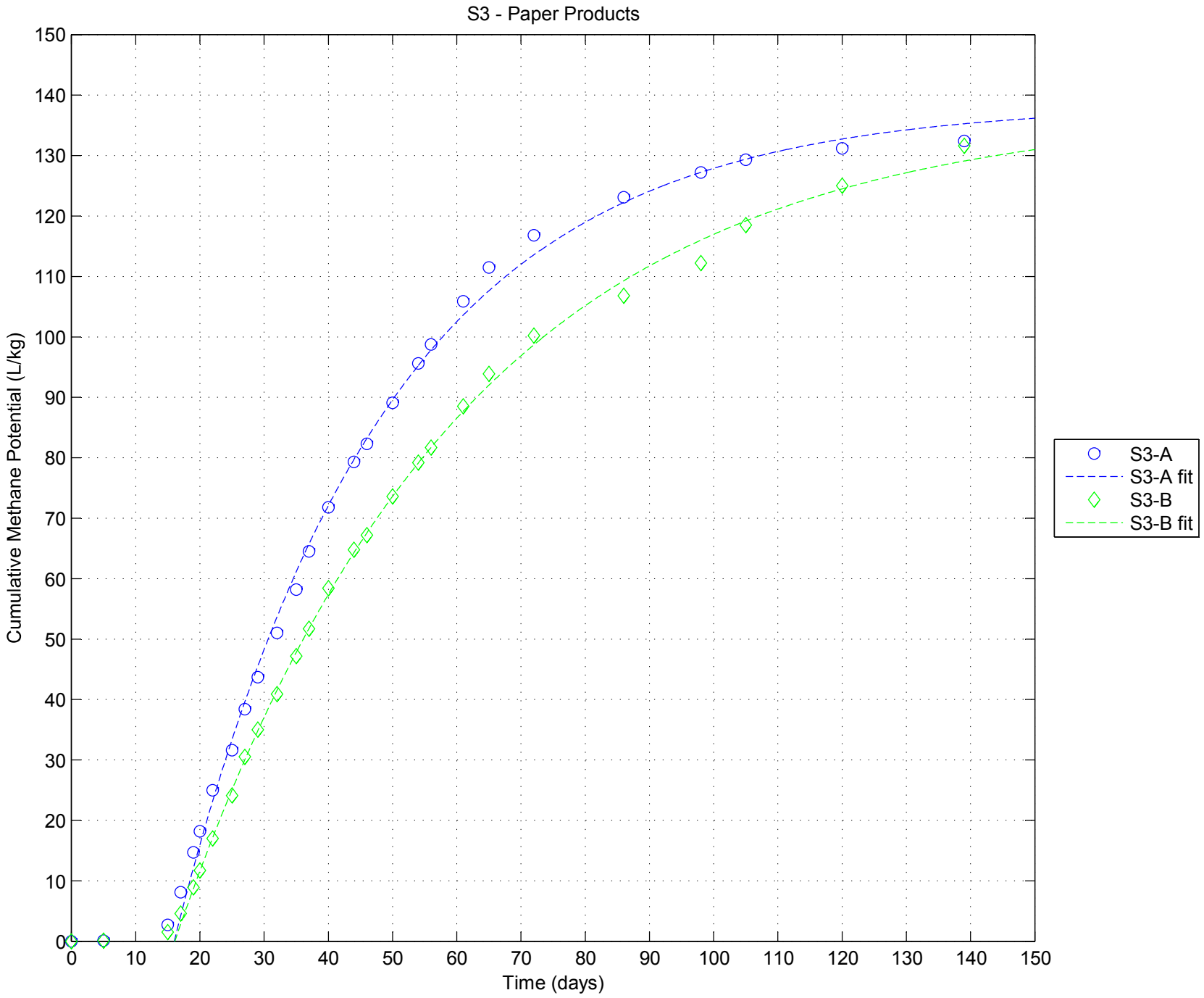

BH18
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH18A-A
BH18A-A fit
BH18A-B
BH18A-B fit
BH18A-C
BH18A-C fit
BH18B-A
BH18B-A fit
BH18B-B
BH18B-B fit
BH18B-C
BH18B-C fit

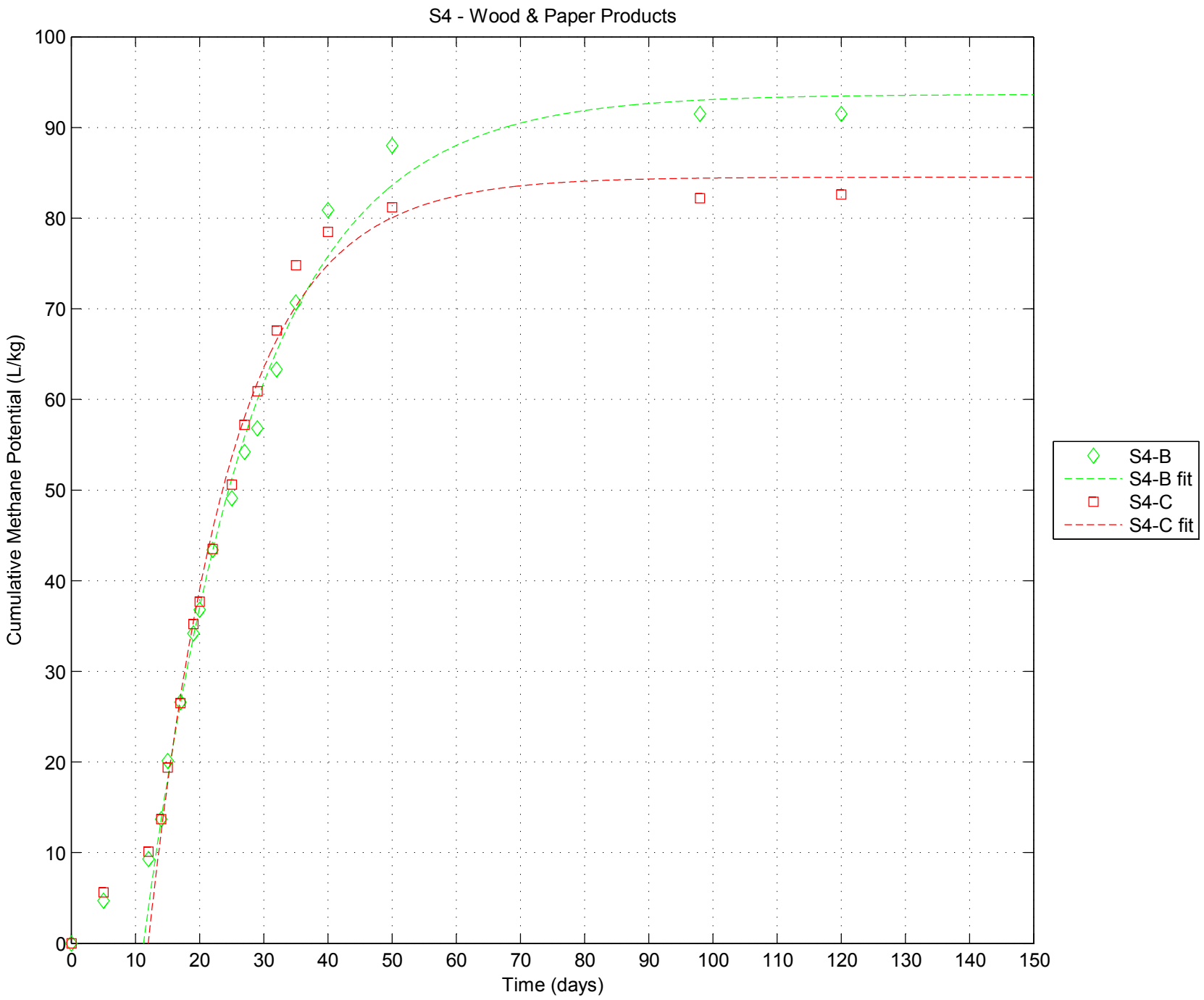

BH19A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH19A-A
BH19A-A fit
BH19A-B
BH19A-B fit
BH19A-C
BH19A-C fit

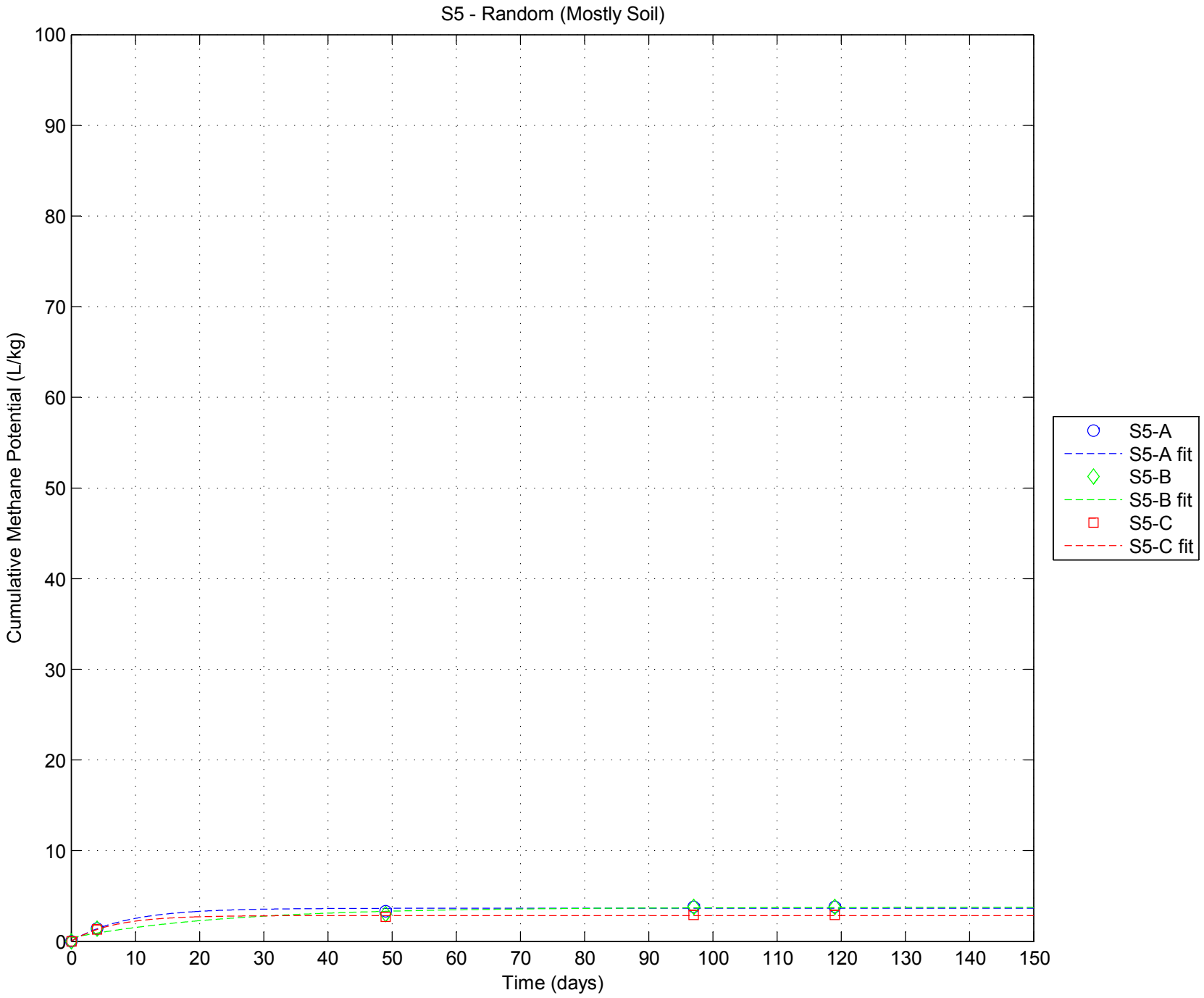

BH20A
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH20A-A
BH20A-A fit
BH20A-B
BH20A-B fit
BH20A-C
BH20A-C fit

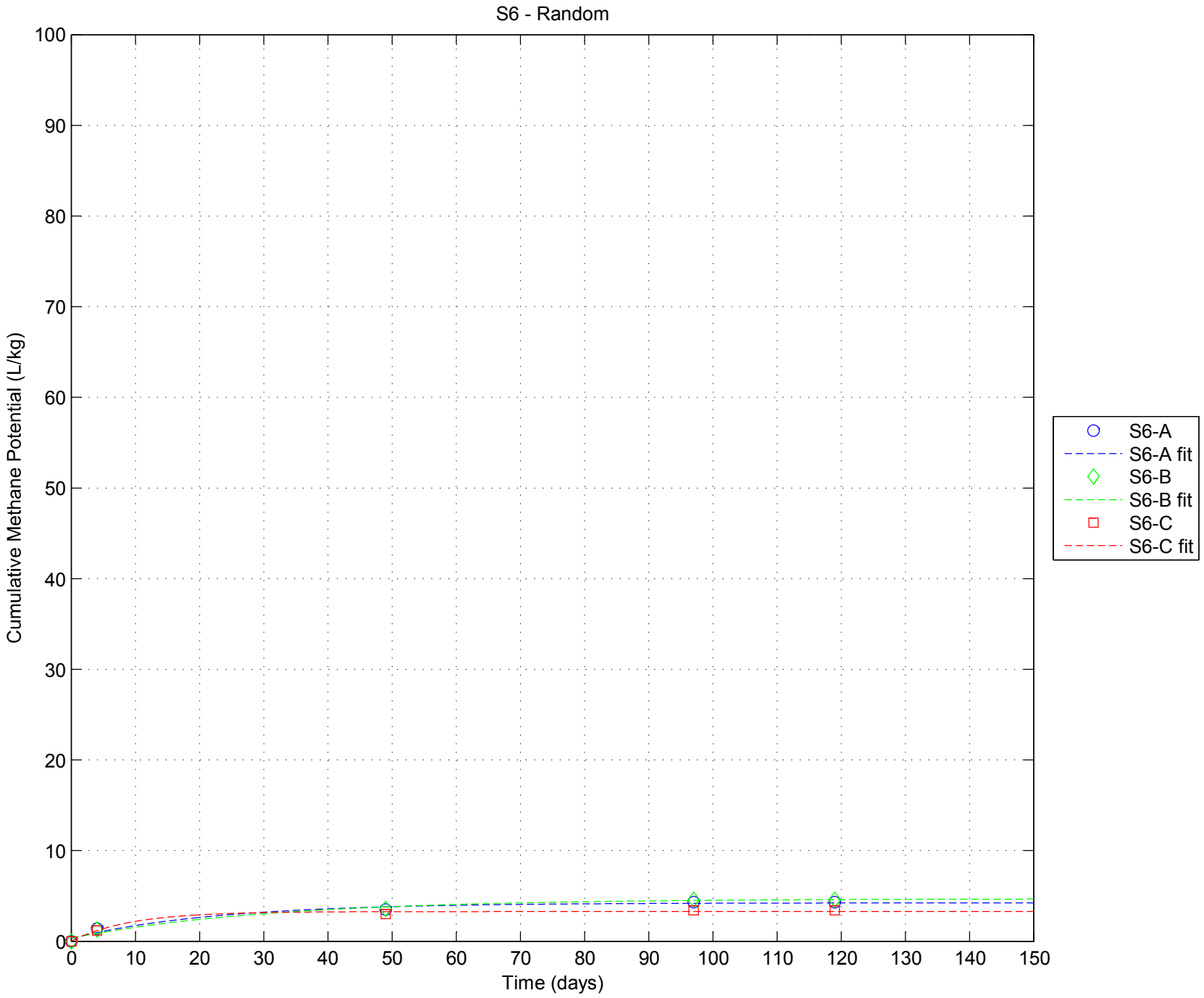

S1 - Wood & Paper Products
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
S1-A
S1-A fit (Stage 1)
S1-A fit (Stage 2)
S1-B
S1-B fit
S1-C
S1-C fit (Stage 1)
S1-C fit (Stage 2)

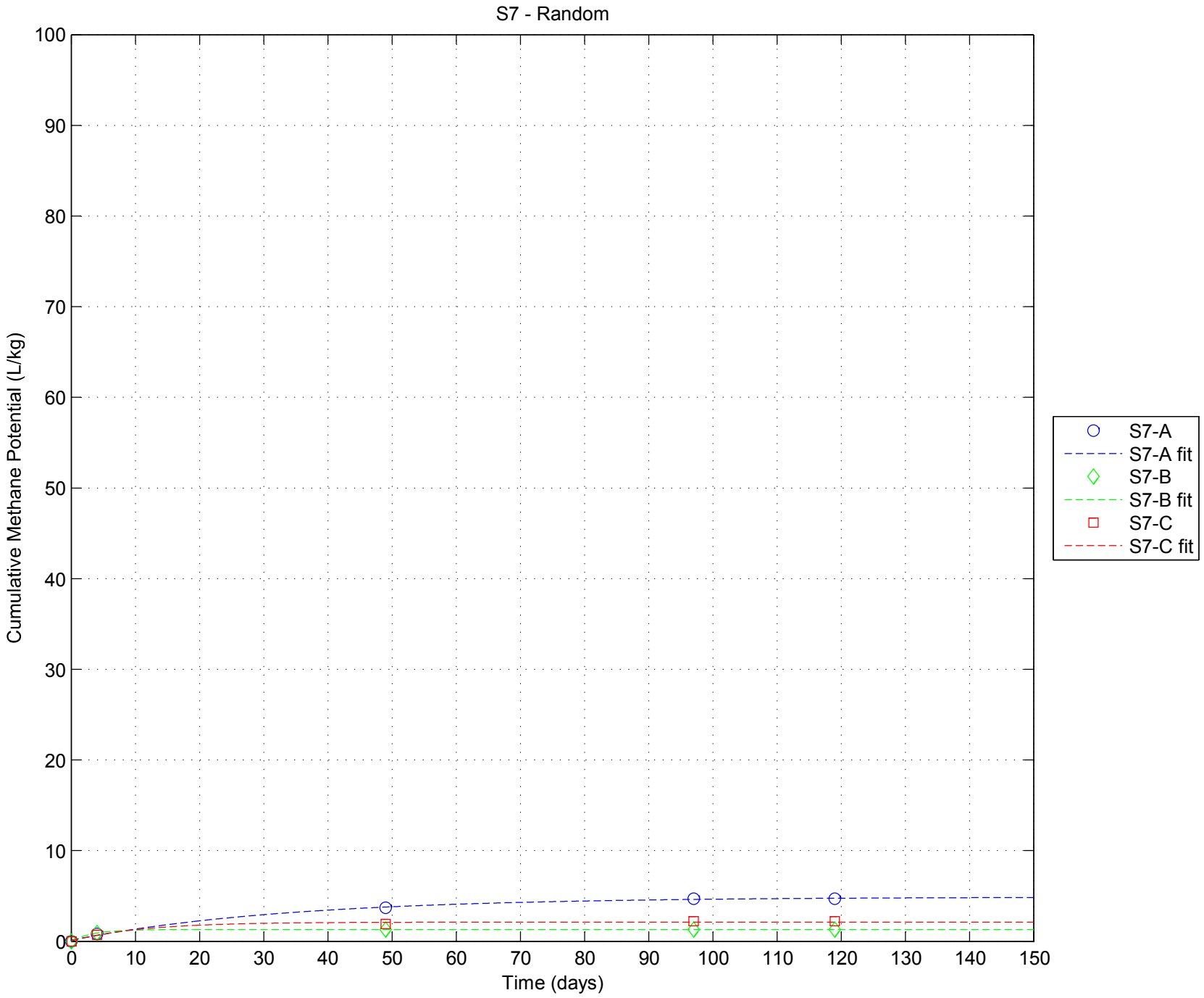

S2 - Soil & Degraded Material
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S2-A
S2-A fit
S2-B
S2-B fit
S2-C
S2-C fit

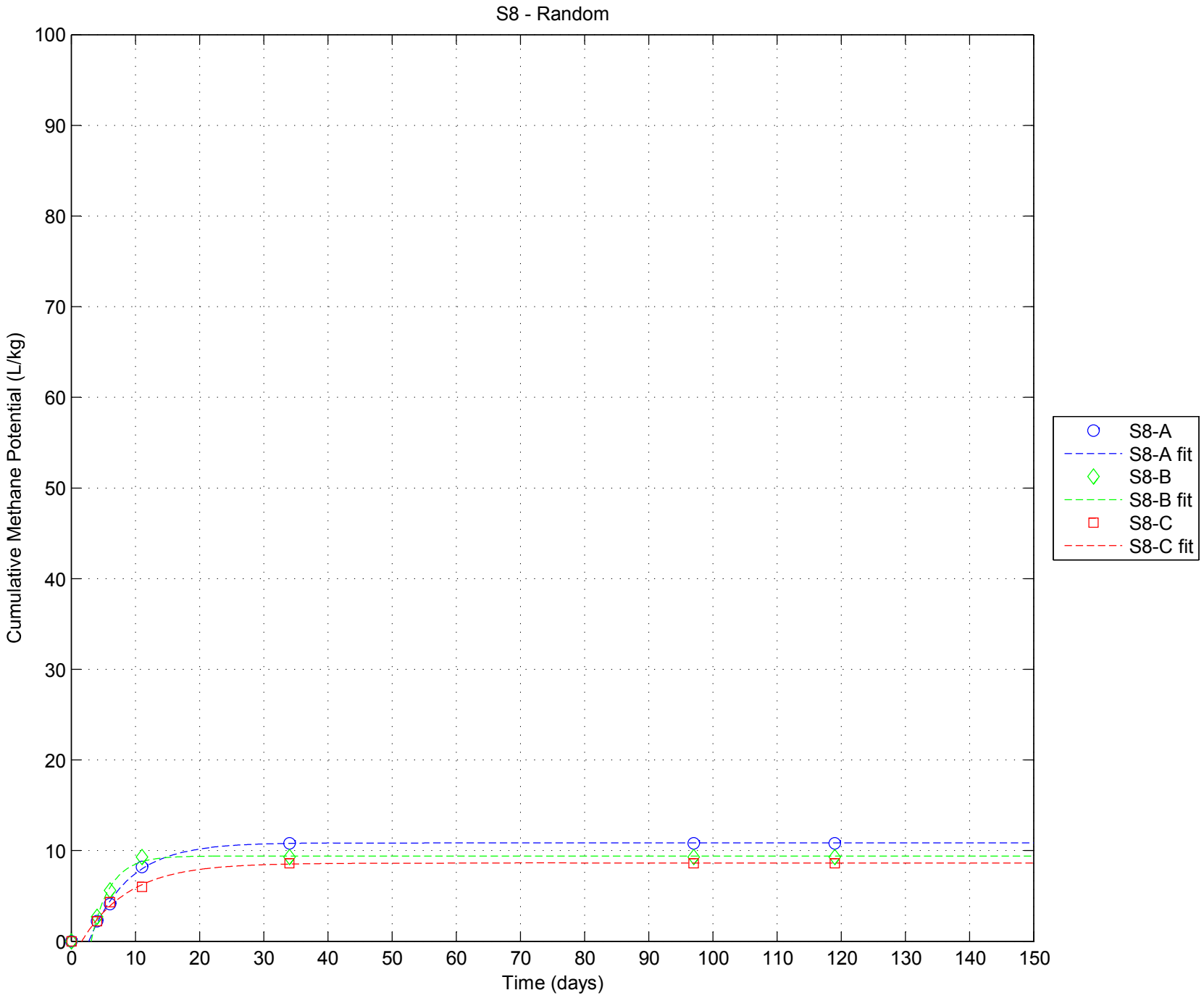

S3 - Paper Products
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S3-A
S3-A fit
S3-B
S3-B fit

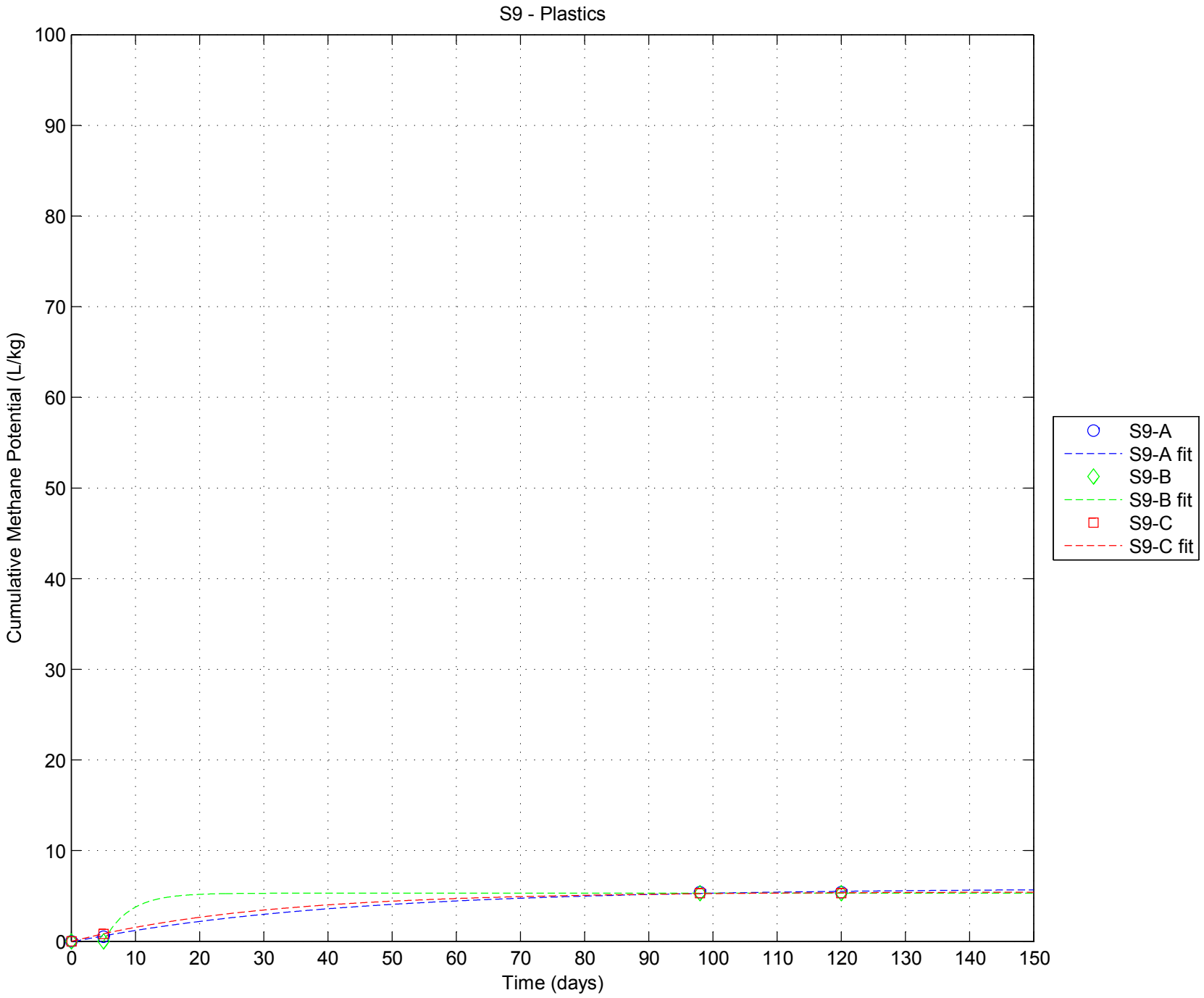

S4 - Wood & Paper Products
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S4-B
S4-B fit
S4-C
S4-C fit

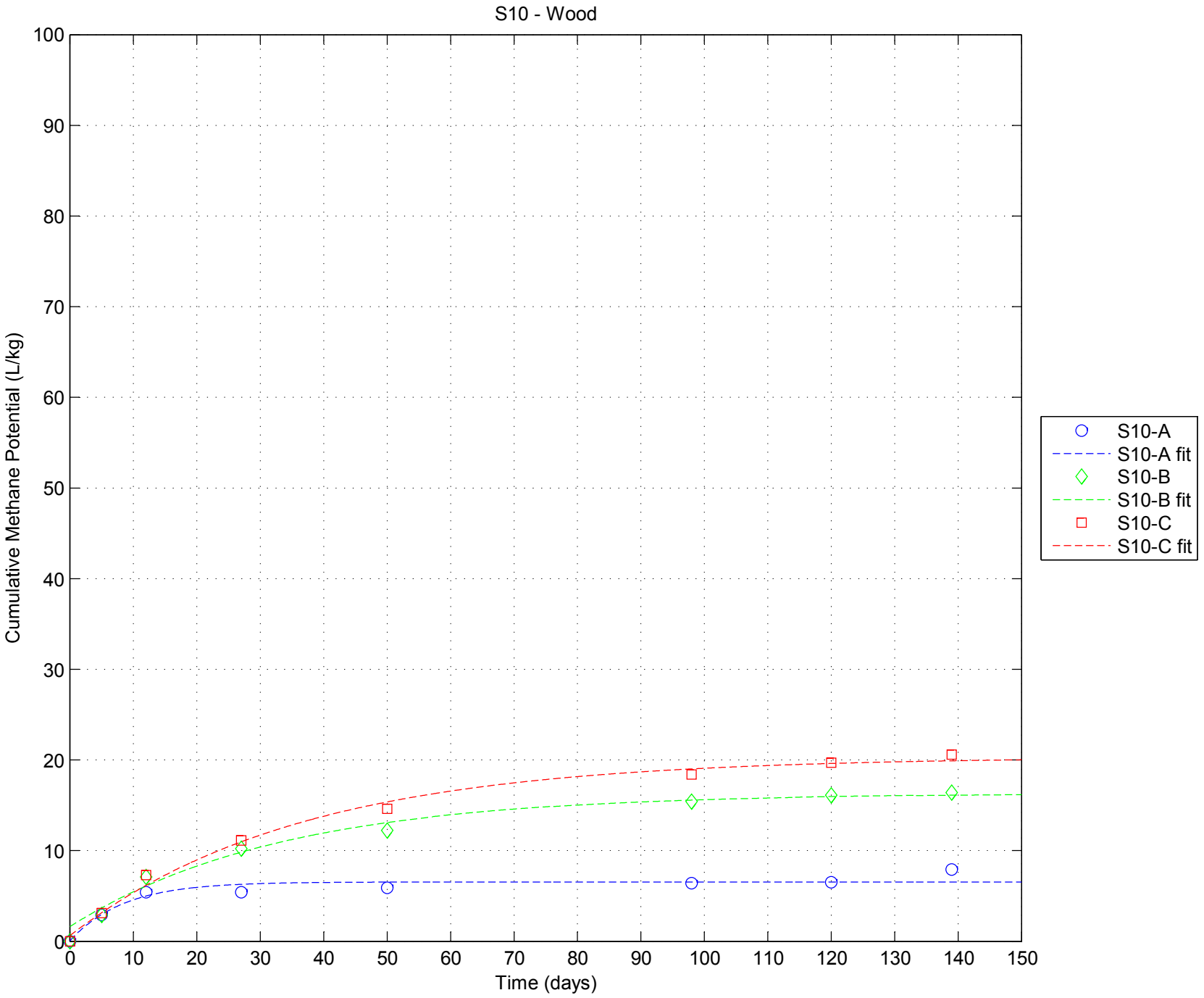

S5 - Random (Mostly Soil)
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S5-A
S5-A fit
S5-B
S5-B fit
S5-C
S5-C fit

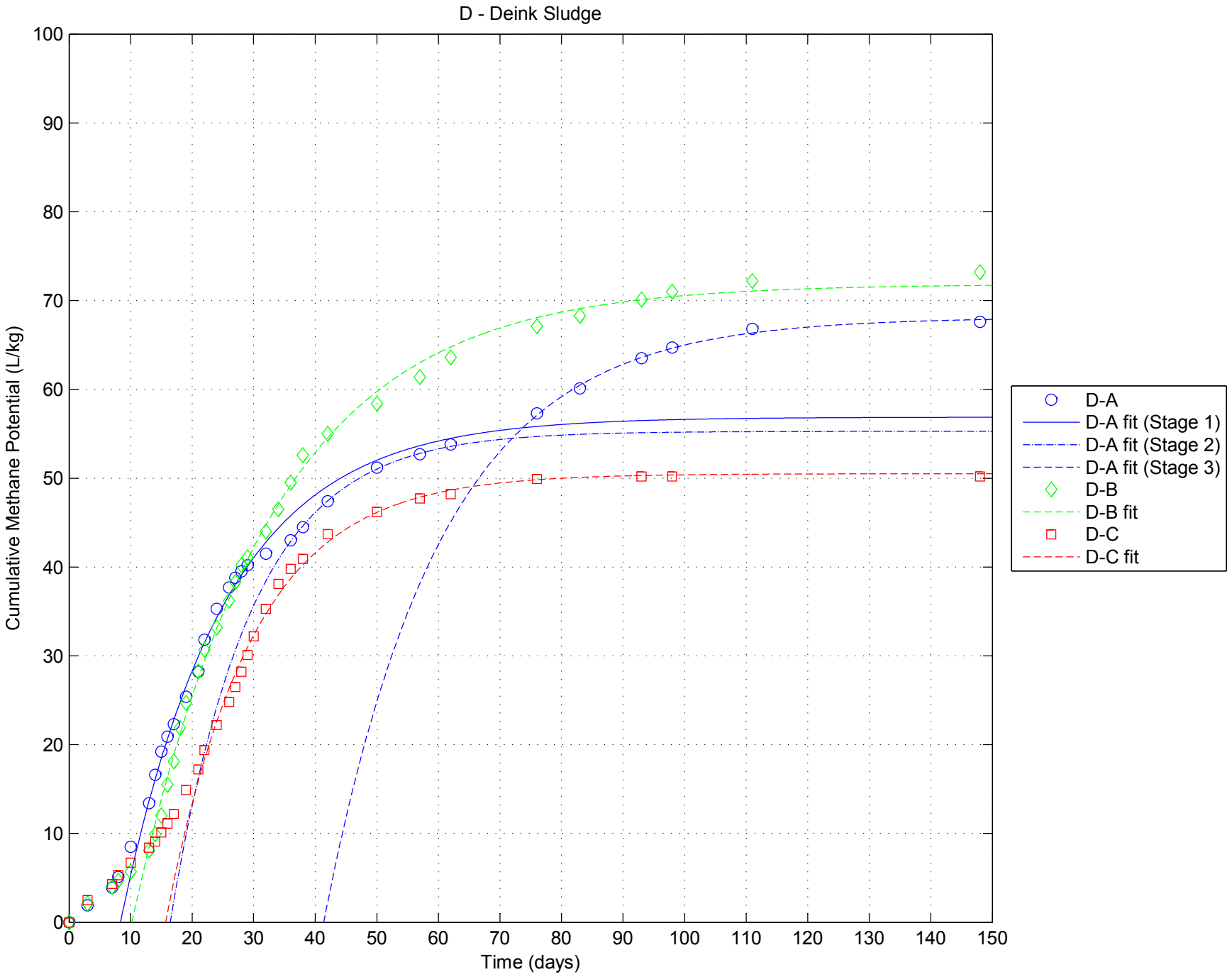

S6 - Random
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S6-A
S6-A fit
S6-B
S6-B fit
S6-C
S6-C fit

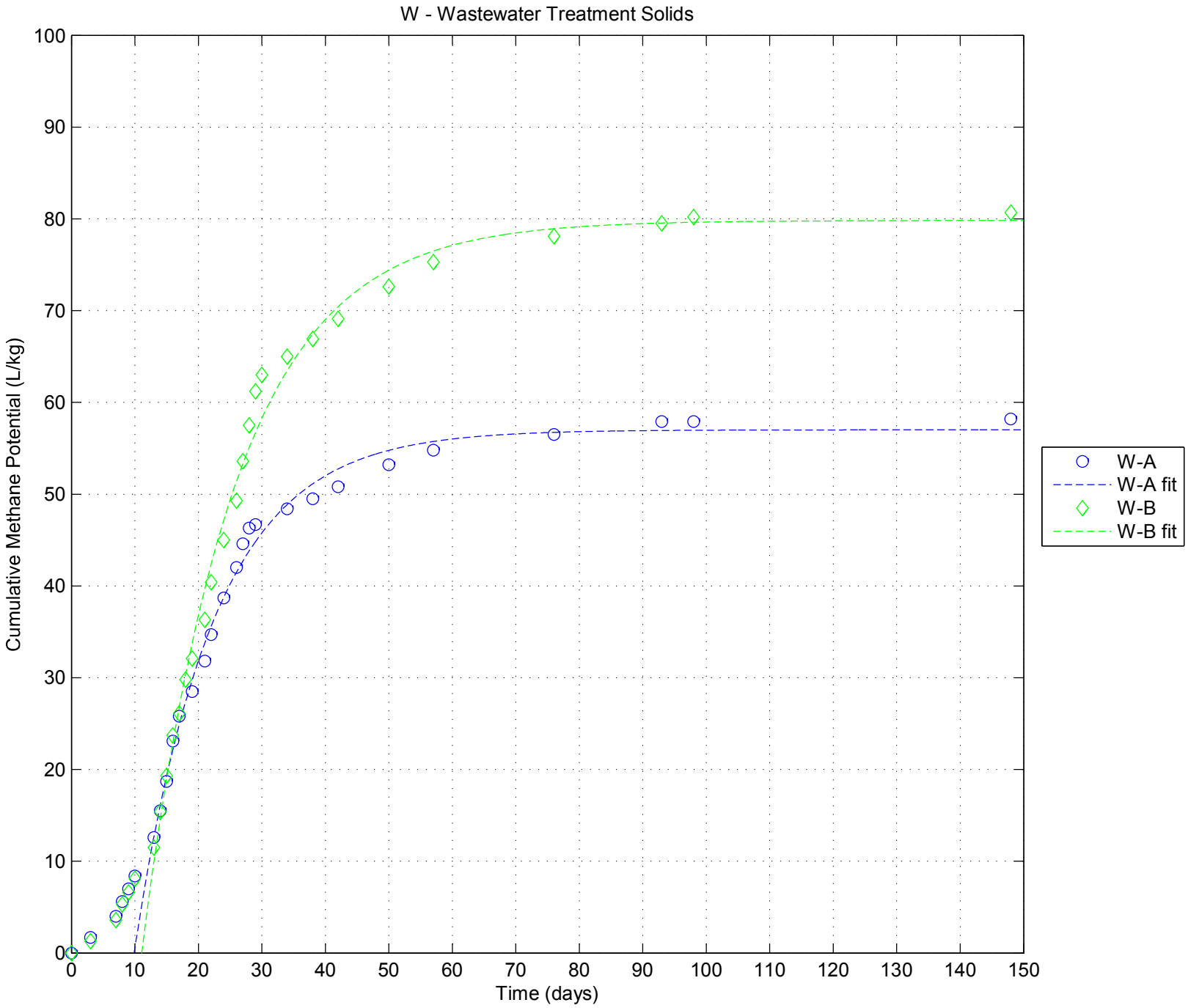

S7 - Random
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S7-A
S7-A fit
S7-B
S7-B fit
S7-C
S7-C fit

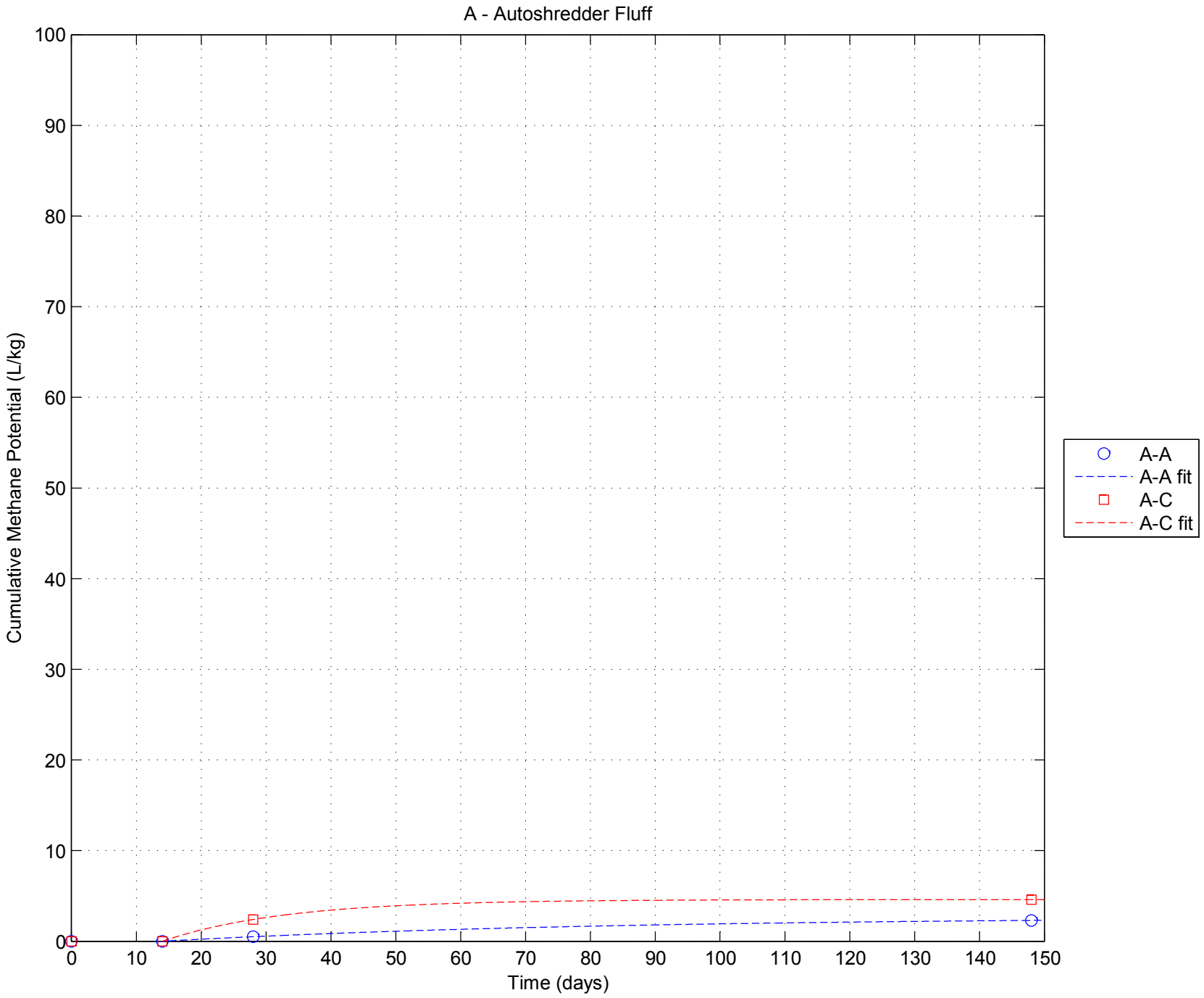

S8 - Random
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S8-A
S8-A fit
S8-B
S8-B fit
S8-C
S8-C fit

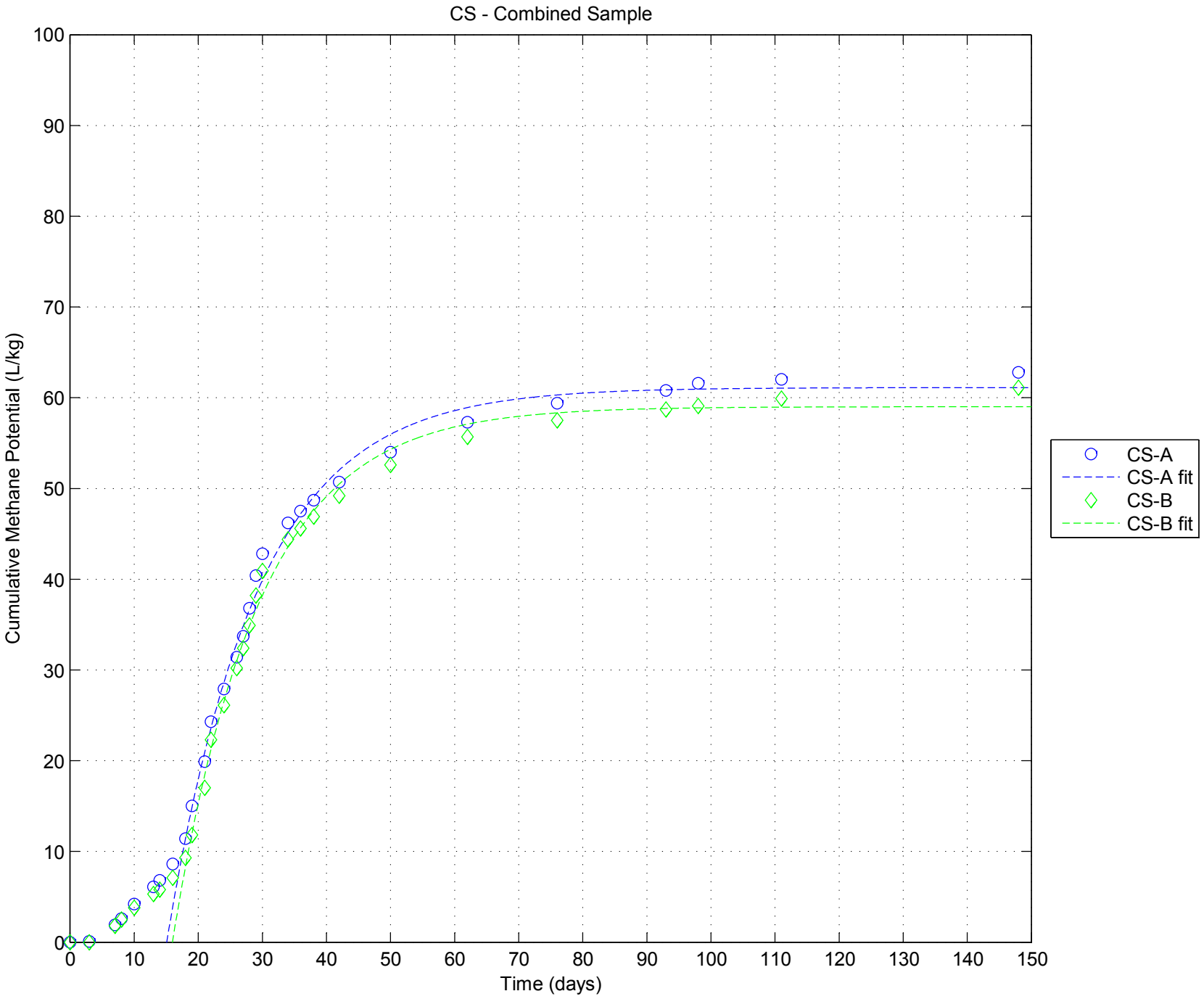

S9 - Plastics
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S9-A
S9-A fit
S9-B
S9-B fit
S9-C
S9-C fit

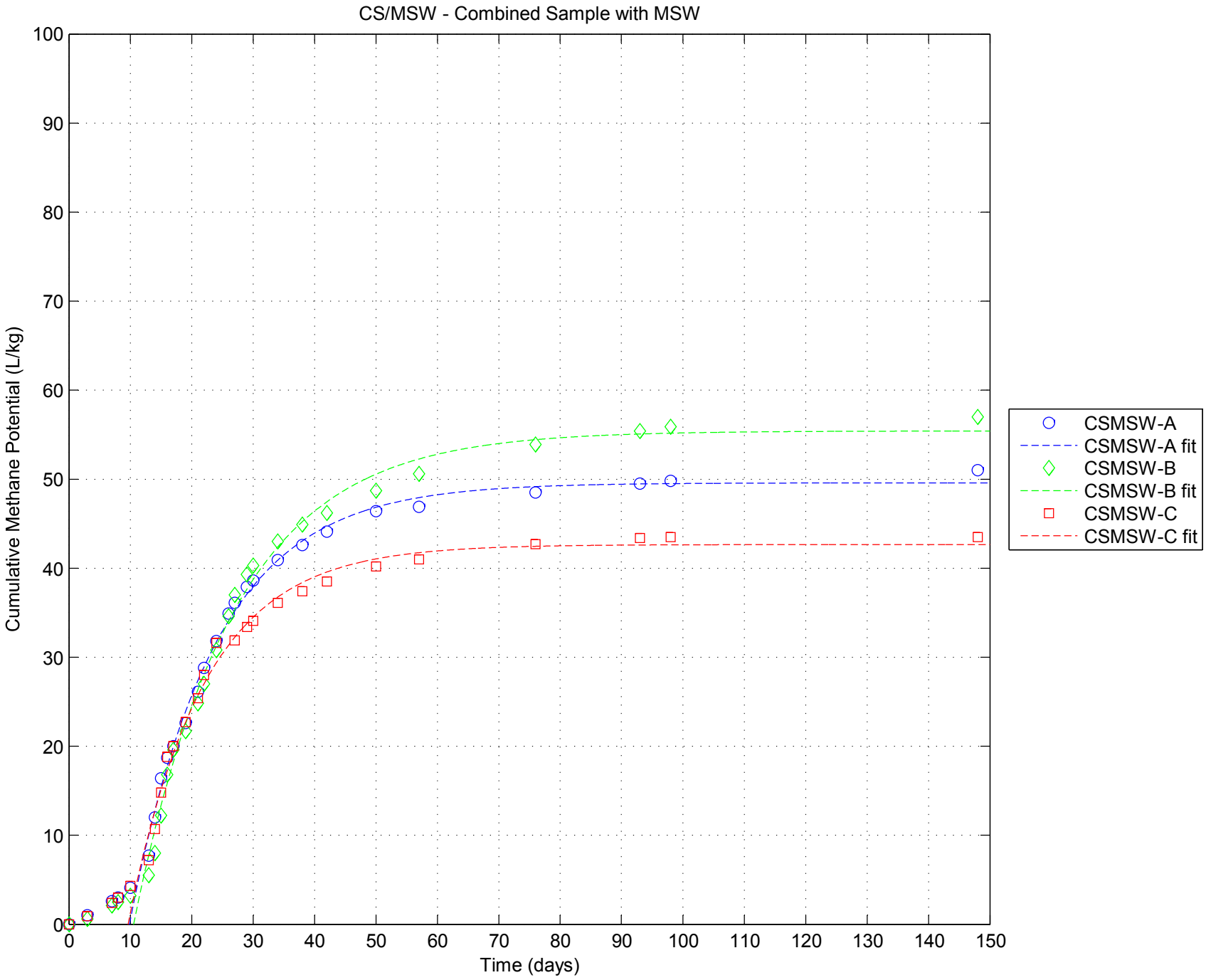

S10 - Wood
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S10-A
S10-A fit
S10-B
S10-B fit
S10-C
S10-C fit


D - Deink Sludge
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
D-A
D-A fit (Stage 1)
D-A fit (Stage 2)
D-A fit (Stage 3)
D-B
D-B fit
D-C
D-C fit


W - Wastewater Treatment Solids
Cumulative Methane Potential (L/kg)
Time (days)
W-A
W-A fit
W-B
W-B fit


A - Autoshredder Fluff
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
A-A
A-A fit
A-C
A-C fit


CS - Combined Sample
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
CS-A
CS-A fit
CS-B
CS-B fit


CS/MSW - Combined Sample with MSW
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
CSMSW-A
CSMSW-A fit
CSMSW-B
CSMSW-B fit
CSMSW-C
CSMSW-C fit

Appendix C

Curve Fit Parameters

Sample	Fit	BMP_{ult}	k	t_{lag}	Included Range of t
M14	A	6.83	0.09659	0.1355	0-26
M14	B	8.432	0.07589	1.63	4-26
M14	C	12.65	0.08222	2.911	4-26
M15	B	6.763	0.2176	2.512	4-15
M15	C	6.171	0.2046	2.426	4-15
M17	A	6.112	0.1203	11.27	13-26
M17	B	7.278	0.1164	9.446	13-26
M17	C	8.939	0.09589	11.52	13-26
BH1A	A	34.34	0.05413	7.349	11-130
BH1A	B	31.57	0.05233	6.73	11-130
BH1A	C	30.82	0.05205	6.983	9-130
BH2A	A (Stage 1)	11.98	0.1578	9.598	14-21
BH2A	A (Stage 2)	22.67	0.1416	25.000	34-98
BH2A	B (Stage 1)	13.31	0.1148	9.127	14-21
BH2A	B (Stage 2)	24.8	0.1283	26.56	34-98
BH2A	C (Stage 1)	11.25	0.2178	8.15	9-14
BH2A	C (Stage 2)	31.45	0.05263	20.47	31-98
BH2B	A	25.84	0.03608	3.912	6-94
BH2B	B	29.42	0.04556	6.131	13-94
BH2B	C	32.73	0.03091	4.873	6-94
BH2B	D	20.04	0.05772	5.239	6-94
BH3A	A	10.18	0.06051	6.497	12-98
BH3A	B	11.58	0.04454	5.233	12-98
BH3A	C	9.612	0.05284	4.779	18-98
BH4A	A	6.482	0.04131	1.584	5-98
BH4A	B	6.282	0.0516	2.267	5-98
BH4A	C	7.367	0.03815	1.161	5-98
BH5A	A	11.46	0.03333	2.664	5-98
BH5A	B	13.29	0.02788	2.156	5-98
BH5A	C	13.38	0.0323	2.391	5-98
BH6A	A	25.1	0.05258	0.9291	8-142
BH6A	B	26.53	0.04846	1.966	3-142
BH6A	C	26.63	0.05347	2.379	3-142
BH7A	A	46.14	0.05069	10.58	21-142
BH7A	B	45.15	0.04157	5.619	8-142
BH7A	C	45.83	0.04642	5.96	8-142
BH8A	A (Stage 1)	8.963	0.2232	2.281	3-15
BH8A	A (Stage 2)	24.65	0.02063	-4.748	28-142
BH8A	B (Stage 1)	8.69	0.2715	2.979	5-15
BH8A	B (Stage 2)	17.92	0.0896	16.94	28-142
BH8A	C (Stage 1)	9.209	0.2527	3.023	5-15
BH8A	C (Stage 2)	24.41	0.0406	11.04	28-142

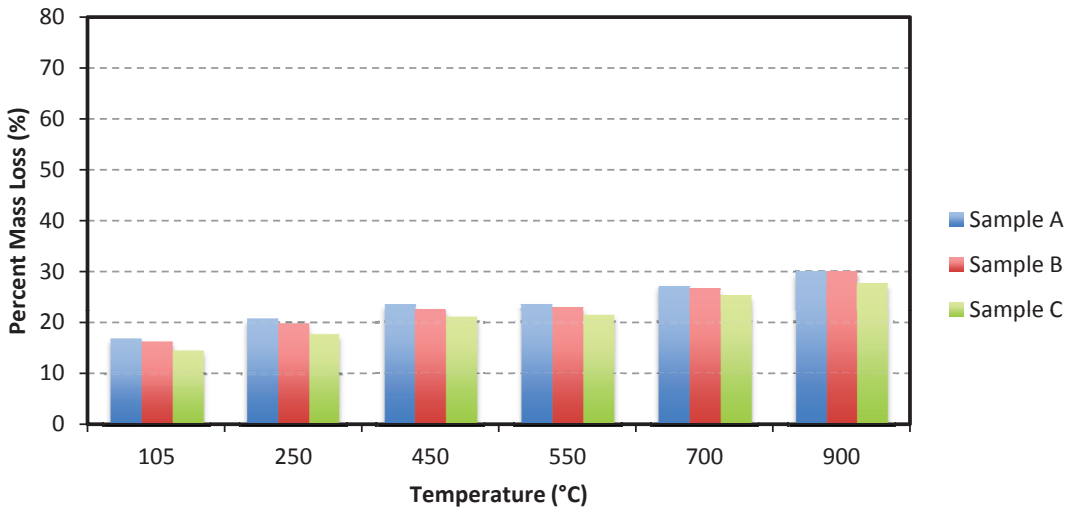
Sample	Fit	BMP_{ult}	k	t_{lag}	Included Range of t
BH9B	A (Stage 1)	11.36	0.1239	4.119	5-16
BH9B	A (Stage 2)	38.26	0.05537	16.13	26-148
BH9B	B (Stage 1)	8.783	0.1402	4.621	7-16
BH9B	B (Stage 2)	43.82	0.03993	16.48	26-148
BH10A	A	28.39	0.08785	2.539	5-92
BH10A	B	32.61	0.08691	4.444	8-92
BH10A	D	29.42	0.08698	5.284	8-94
BH10A	E	28.98	0.08807	5.237	8-94
BH10A	F	26.63	0.09723	6.131	8-94
BH11A	A	23.96	0.02937	-4.816	7-107
BH11A	B	26.92	0.0346	-1.169	5-107
BH11A	C	23.23	0.03109	-2.156	5-107
BH12A	A	13.01	0.05053	0.9239	8-113
BH12A	B	12.88	0.05002	5.627	11-113
BH12A	C	14.00	0.06402	4.309	8-113
BH13A	A	36.67	0.04683	6.067	11-148
BH13A	C	40.97	0.03608	5.328	11-148
BH14A	A	5.204	0.05466	-0.8308	0-113
BH14A	B	5.819	0.05136	-0.4792	0-113
BH14A	C	5.669	0.04246	-0.866	0-113
BH15A	A (Stage 1)	7.04	0.3269	3.146	5-11
BH15A	A (Stage 2)	15.96	0.03567	2.364	23-113
BH15A	B	17.9	0.05658	2.152	5-113
BH15A	C (Stage 1)	19.16	0.06941	5.367	8-16
BH15A	C (Stage 2)	17.12	0.02706	-20.81	23-113
BH16A	A	37.52	0.07007	9.68	12-106
BH16A	B	41.08	0.05858	11.37	14-106
BH16A	C	35.7	0.06813	9.825	14-106
BH17A	A	17.36	0.03293	-3.086	5-106
BH17A	B	12.04	0.087	3.469	8-106
BH17A	C	16.91	0.04094	-0.7886	5-106
BH18A	A	5.755	0.0375	1.916	5-71
BH18A	B	6.00	0.02591	-2.844	0-71
BH18A	C	3.925	0.06973	-0.6599	0-71
BH18B	A	2.276	0.009485	-8.163	0-71
BH18B	B	2.848	0.05933	0.173	0-71
BH18B	C	3.356	0.02917	2.4	19-71
BH19A	A	14.15	0.1085	6.854	9-71
BH19A	B	20.52	0.1084	8.243	9-71
BH19A	C	22.39	0.0848	6.771	9-71
BH20A	A	1.2	0.4667	2.95E-06	0-71
BH20A	B	1.4	0.4583	-7.87E-05	0-71
BH20A	C	1.75	0.1861	-3.04E-05	0-71

Sample	Fit	BMP_{ult}	k	t_{lag}	Included Range of t
S1	A (Stage 1)	69.08	0.03777	12.14	14-35
S1	A (Stage 2)	60.9	0.07093	27.67	50-155
S1	B	54.96	0.05799	12.21	14-155
S1	C (Stage 1)	65.99	0.0388	12.69	14-98
S1	C (Stage 2)	76.5	0.1626	126	139-155
S2	A	6.075	0.07878	1.447	5-120
S2	B	7.615	0.03116	-2.962	5-120
S2	C	7.121	0.04018	-1.972	5-120
S3	A	138.5	0.03062	16	19-139
S3	B	137.7	0.02255	16.09	19-139
S4	A	8.734	0.08833	0.009934	0-120
S4	B	93.65	0.05768	11.25	14-120
S4	C	84.52	0.07728	11.96	14-120
S5	A	3.64	0.1184	-0.02595	0-119
S5	B	3.75	0.04107	-2.663	0-119
S5	C	2.834	0.1531	-0.001896	0-119
S6	A	4.24	0.04437	-1.833	0-119
S6	B	4.689	0.03226	-2.356	0-119
S6	C	3.275	0.1105	-0.03604	0-119
S7	A	4.879	0.02981	-0.8161	0-119
S7	B	1.3	0.2947	3.71E-11	0-119
S7	C	2.113	0.09108	-0.1269	0-119
S8	A	10.84	0.1609	2.719	4-119
S8	B	9.405	0.3393	3.056	4-119
S8	C	8.615	0.1373	1.607	4-119
S9	A	5.829	0.02421	0.5526	0-120
S9	B	5.3	0.1159	5	0-120
S9	C	5.451	0.03339	0.09936	0-120
S10	A	6.54	0.1194	-0.03944	0-139
S10	B	16.33	0.03027	-3.434	5-139
S10	C	20.33	0.02758	-1.109	0-139
D	A (Stage 1)	56.87	0.05912	8.356	13-32
D	A (Stage 2)	55.28	0.07646	16.44	36-57
D	A (Stage 3)	68.1	0.05262	41.38	76-148
D	B	71.85	0.04478	10.22	15-148
D	C	50.51	0.07137	15.68	21-148
W	A	57.01	0.08075	9.881	15-148
W	B	79.82	0.06917	11.07	13-148
A	A	2.665	0.01484	14	14-148
A	C	4.604	0.05262	14	14-148
CS	A	61.12	0.07083	15.08	18-148
CS	B	59.01	0.07431	15.96	21-148
CS/MSW	A	49.59	0.07204	9.836	14-148
CS/MSW	B	55.42	0.06166	10.52	15-148
CS/MSW	C	42.66	0.08109	9.605	14-148

Appendix D

LOI Plots

M13


80
70
60
50
40
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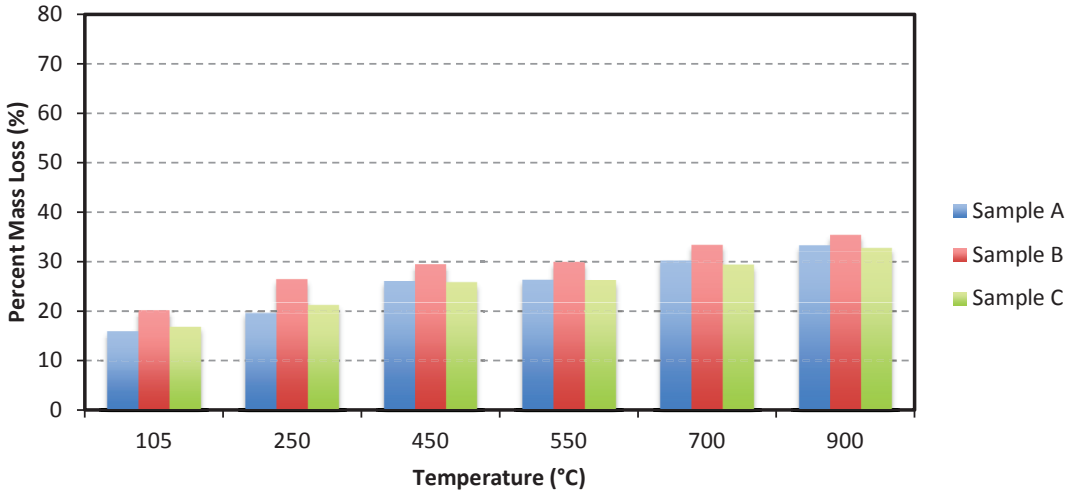
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

M14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60
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Percent Mass Loss (%)
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250
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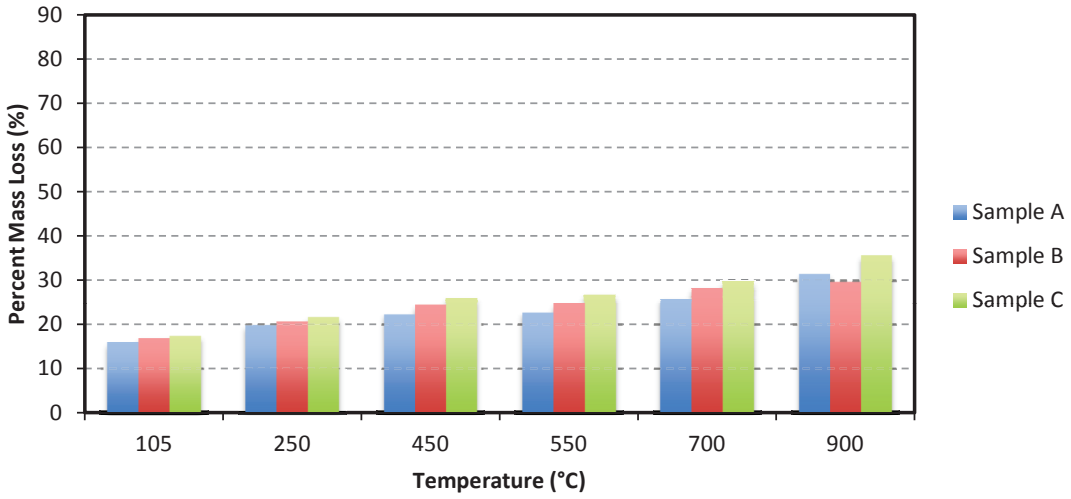
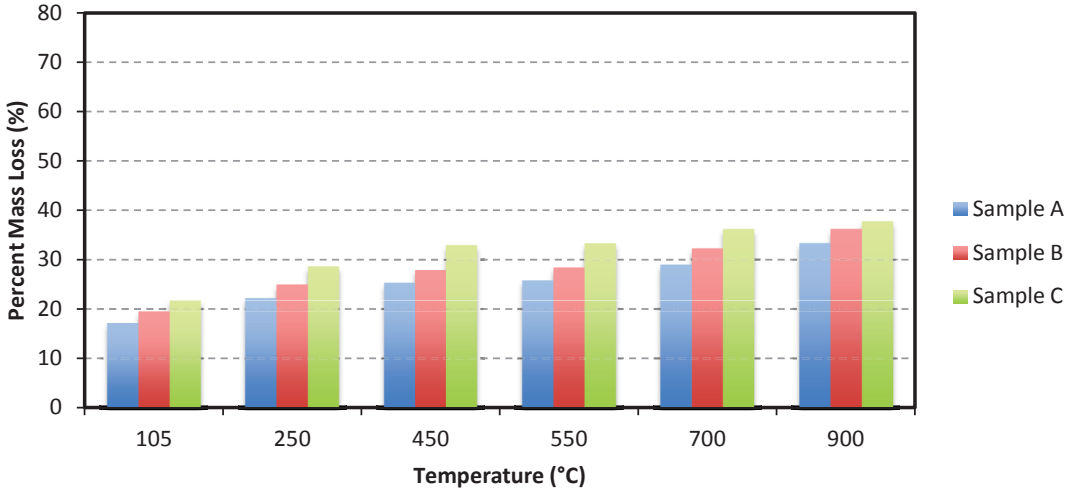
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

M15


90
80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

M17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70
60
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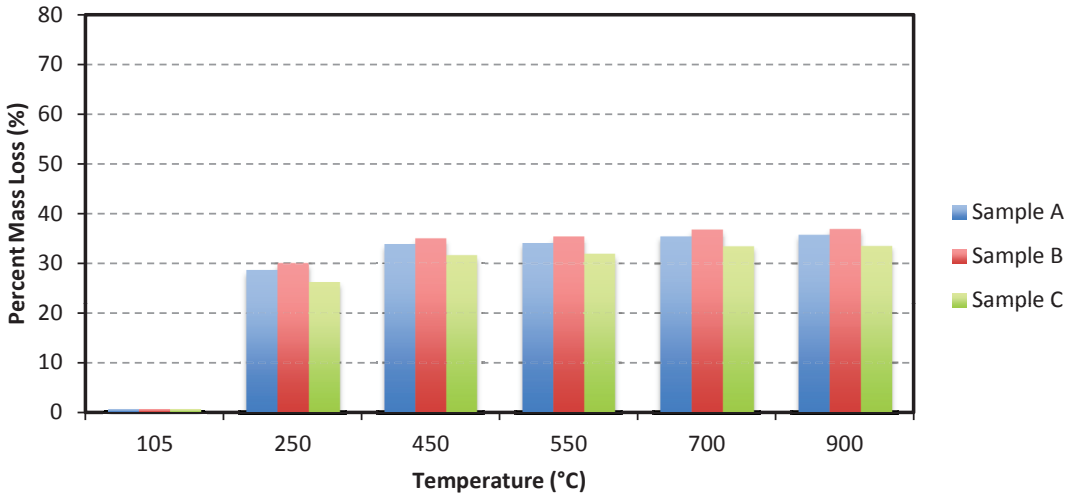
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH1A


80
70
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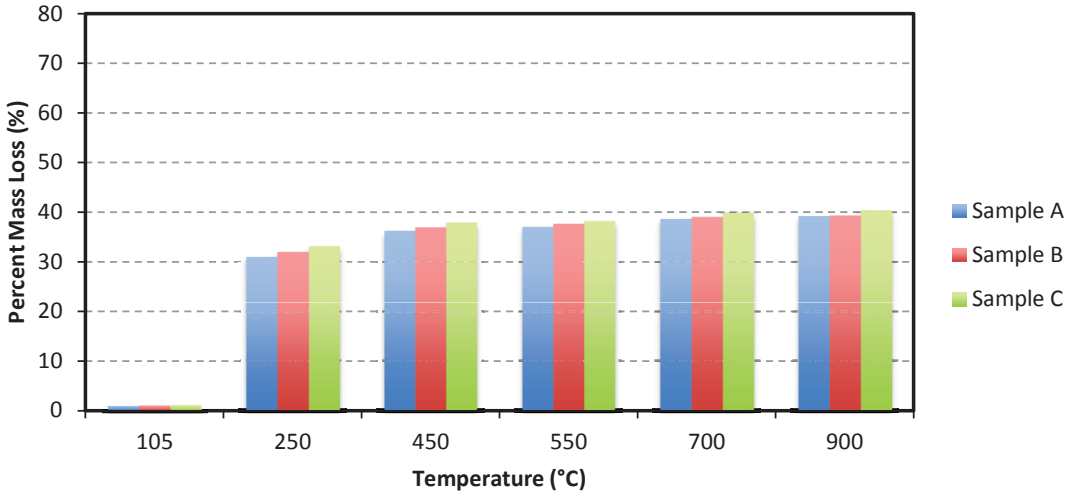
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH2B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70
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50
40
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10
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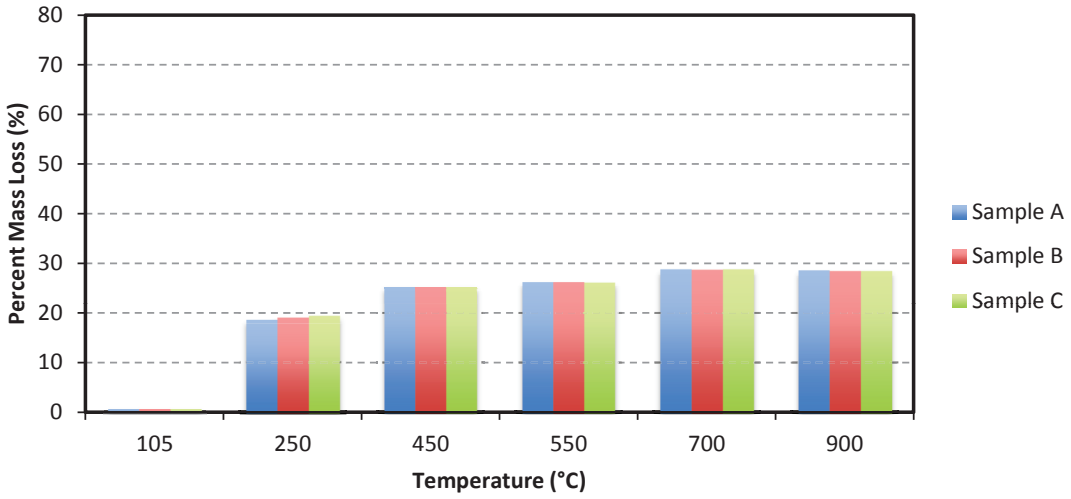
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH3A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70
60
50
40
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20
10
0
Percent Mass Loss (%)
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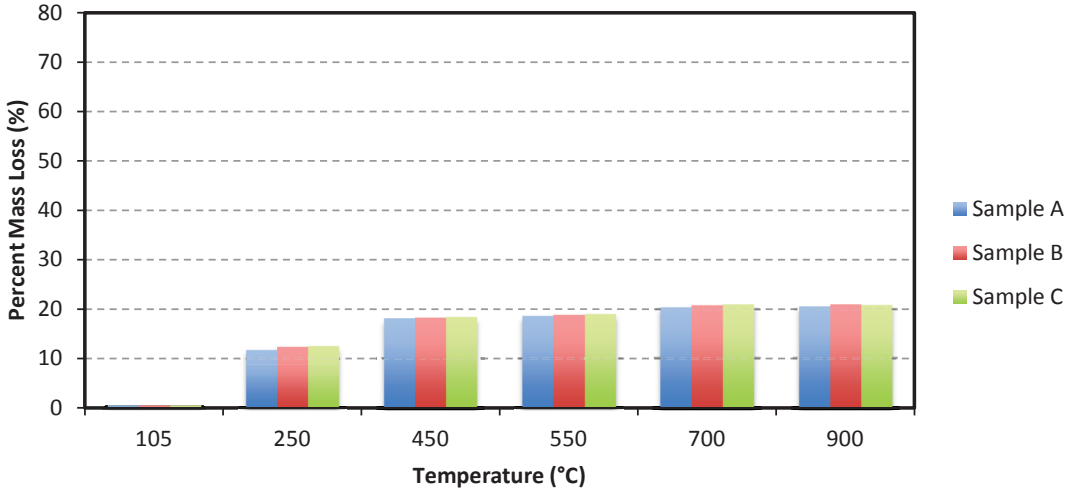
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH4A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70
60
50
40
30
20
10
0
Percent Mass Loss (%)
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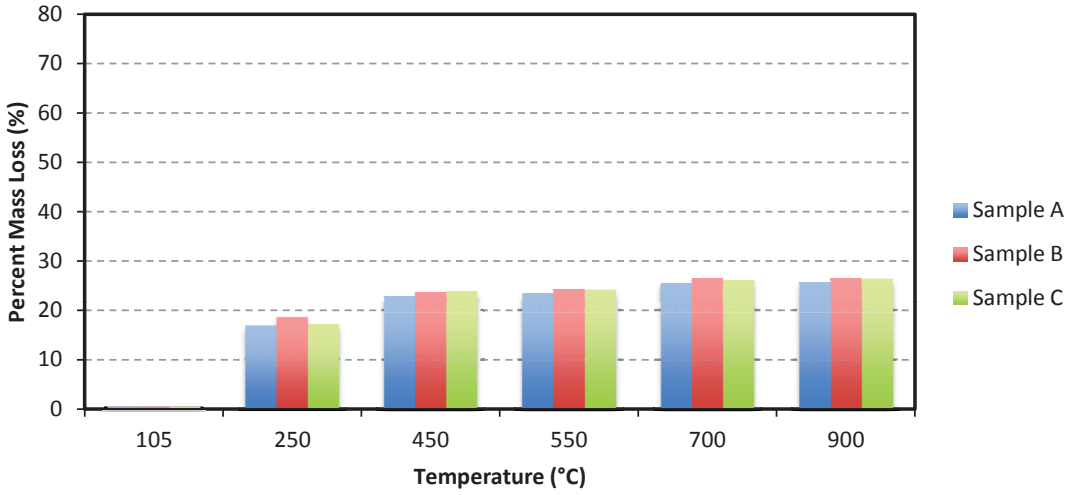
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH5A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40
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20
10
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Percent Mass Loss (%)
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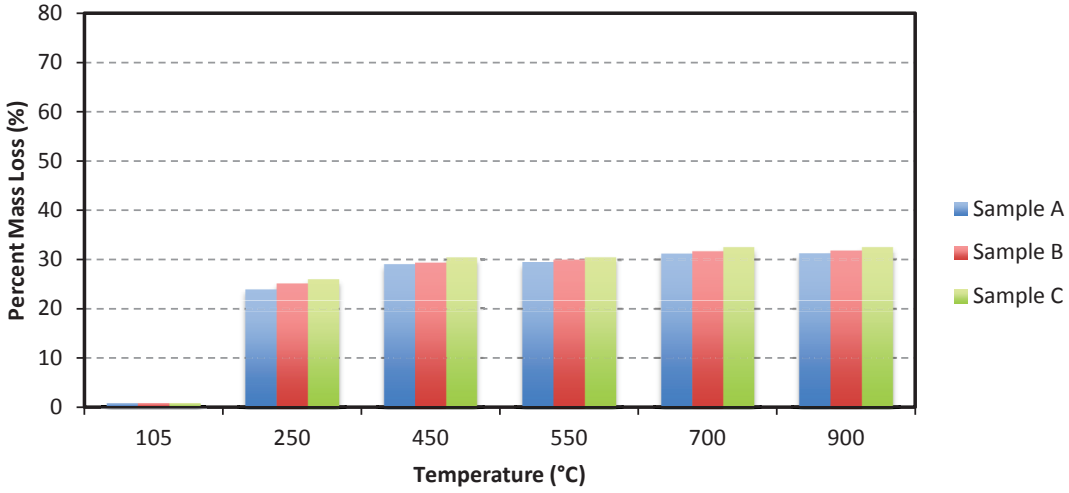
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH6A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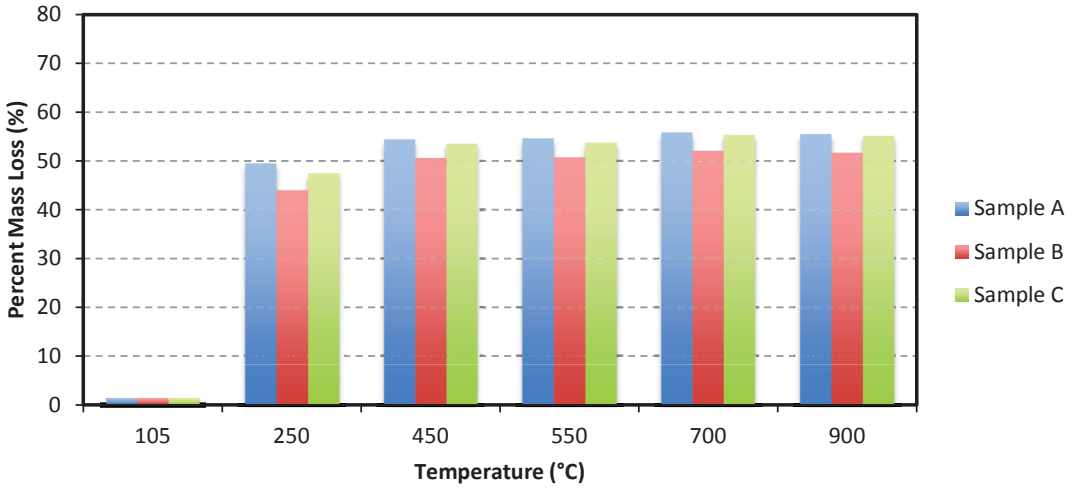
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH7A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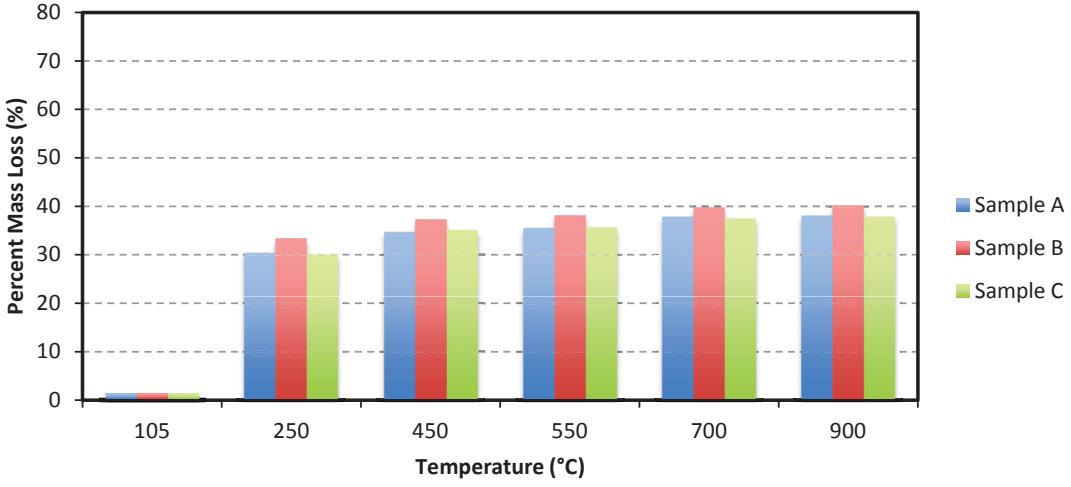
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH8A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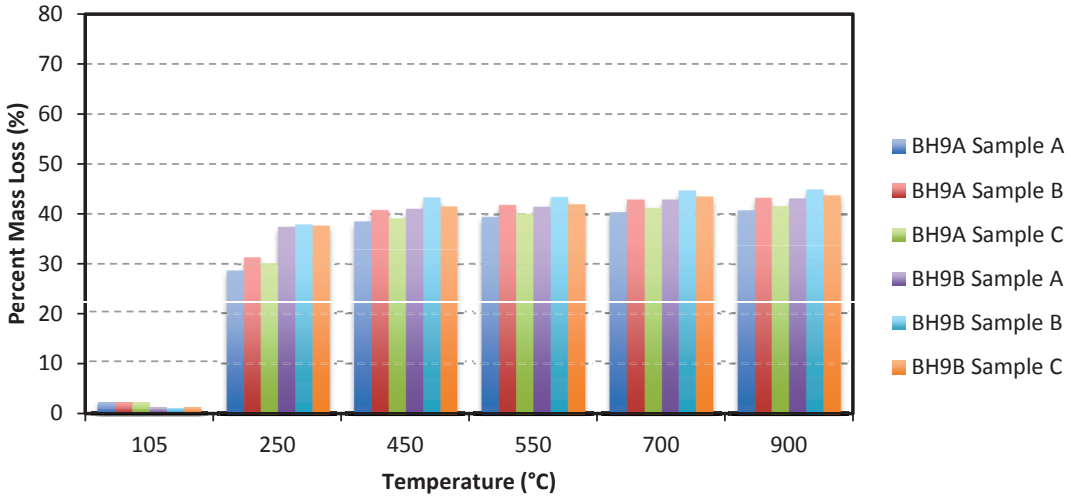
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH9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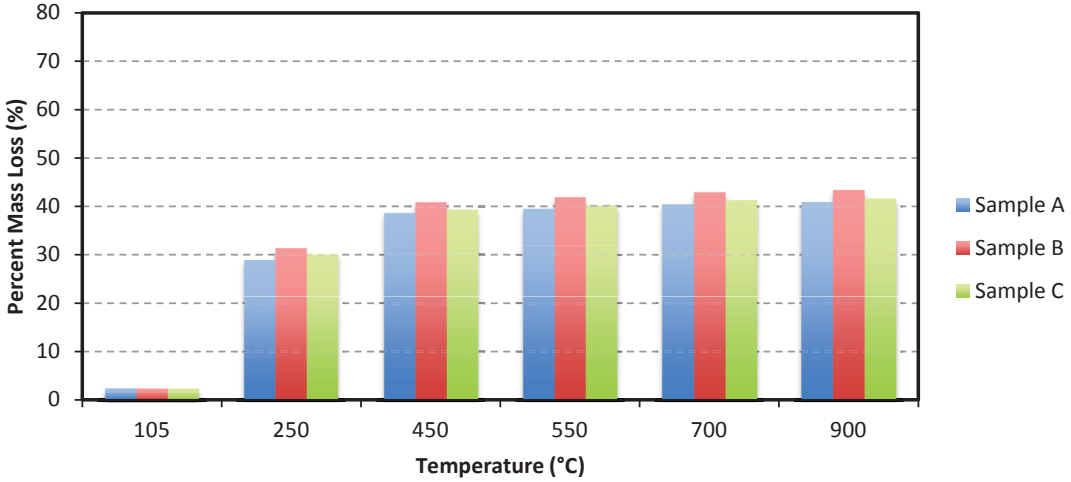
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
BH9A Sample A
BH9A Sample B
BH9A Sample C
BH9B Sample A
BH9B Sample B
BH9B Sample C

BH9A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60
50
40
30
20
10
0
Percent Mass Loss (%)
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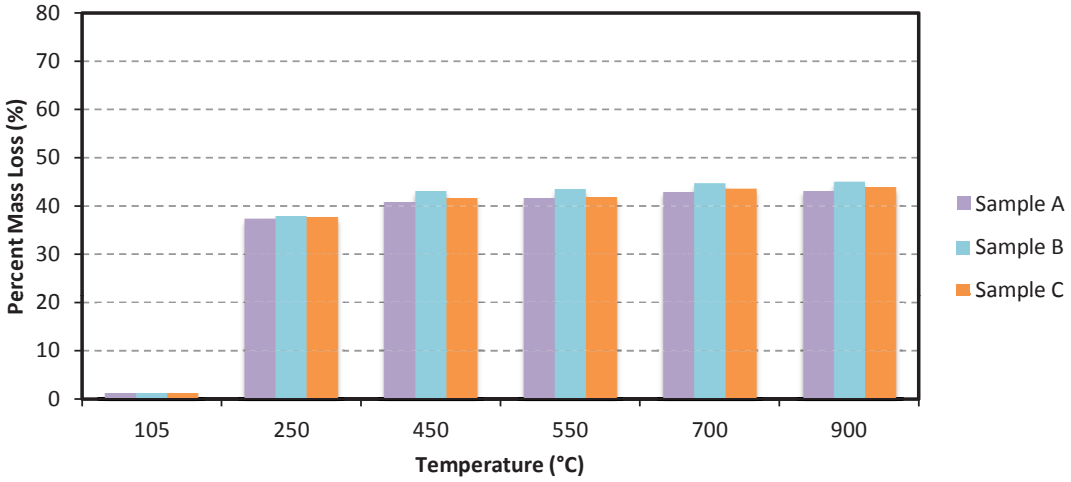
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH9B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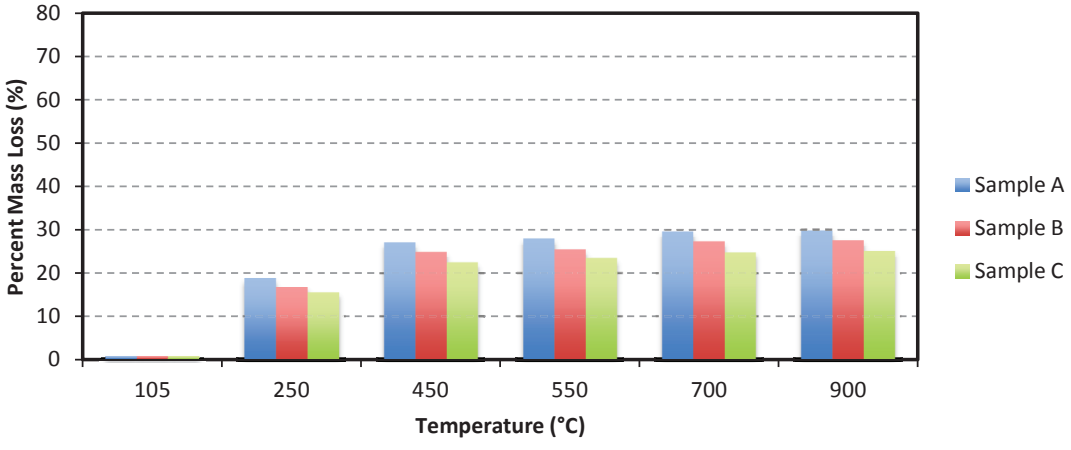
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH10A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60
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40
30
20
10
0
Percent Mass Loss (%)
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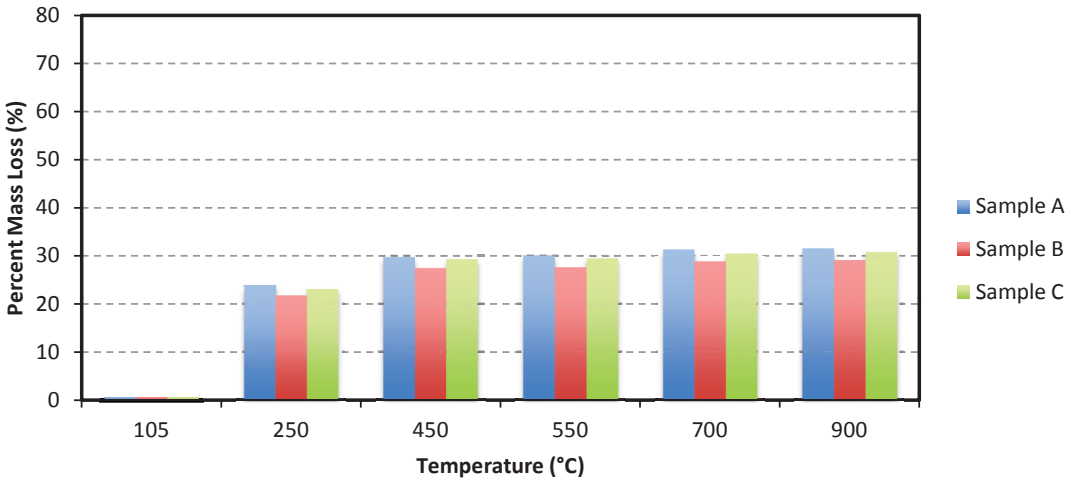
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH11A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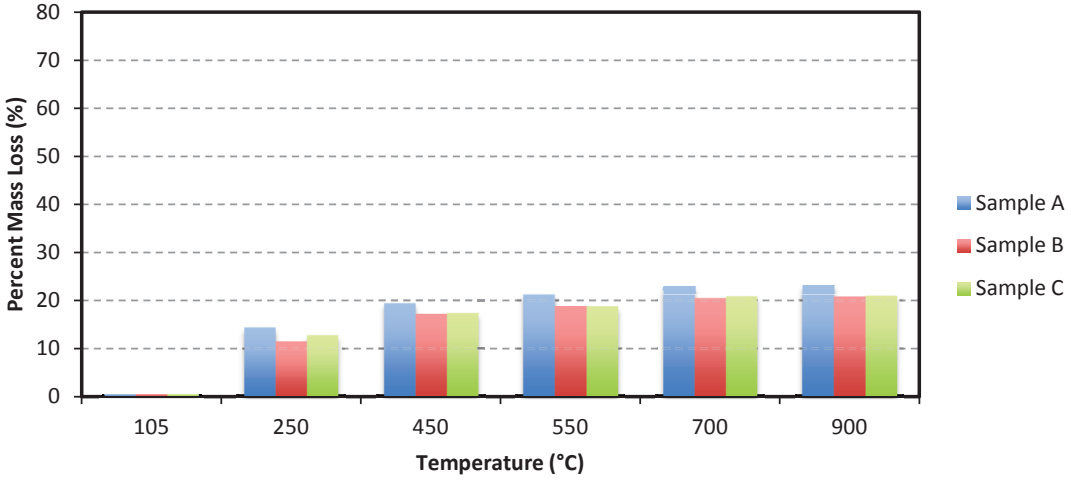
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH12A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60
50
40
30
20
10
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Percent Mass Loss (%)
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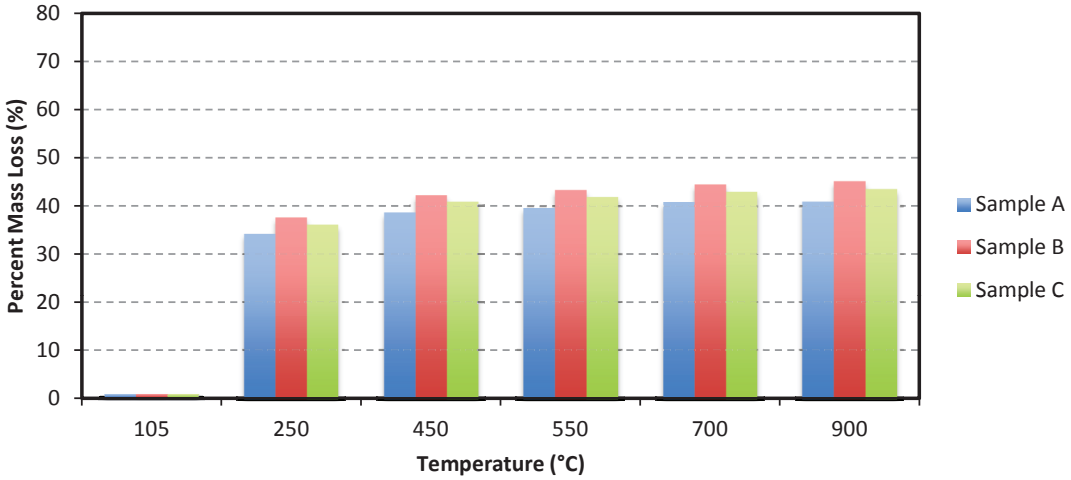
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH13A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70
60
50
40
30
20
10
0
Percent Mass Loss (%)
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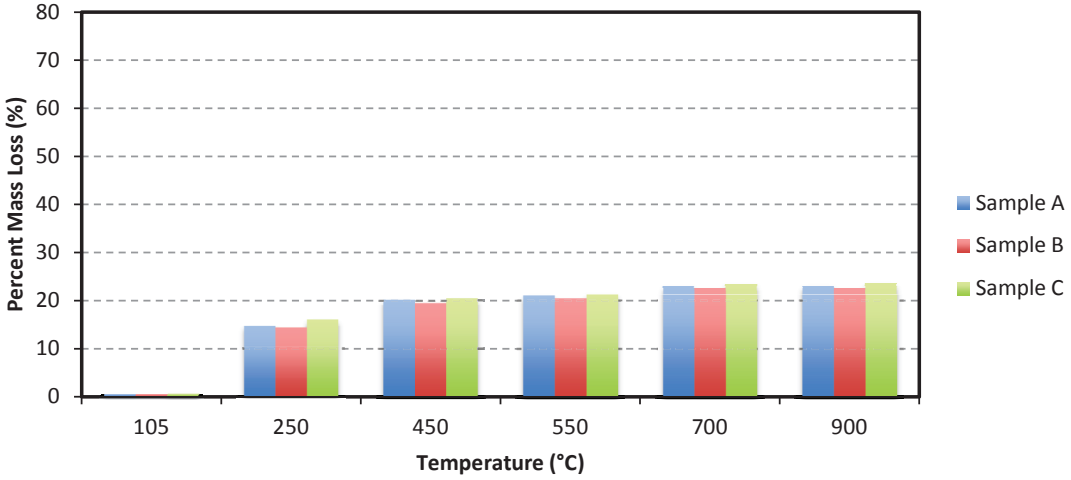
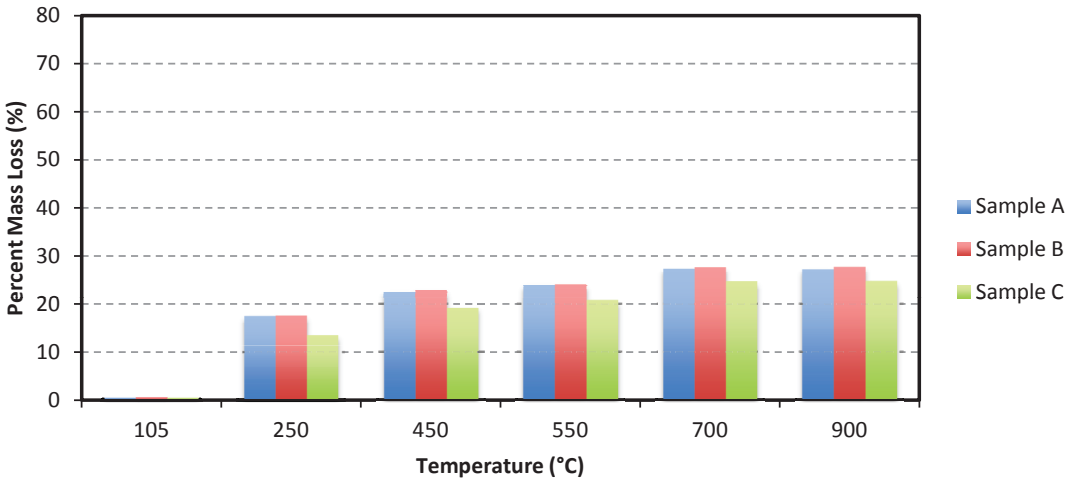
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

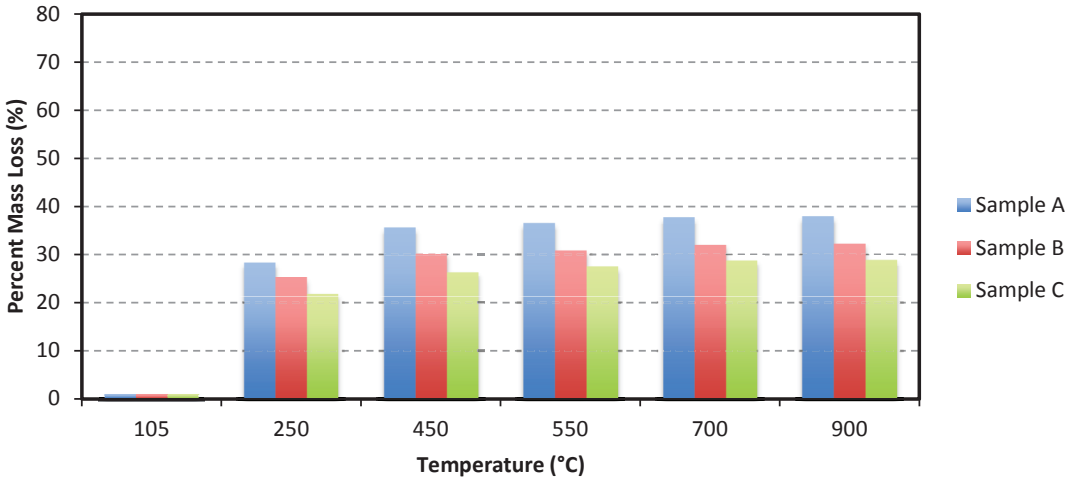
BH14A


80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

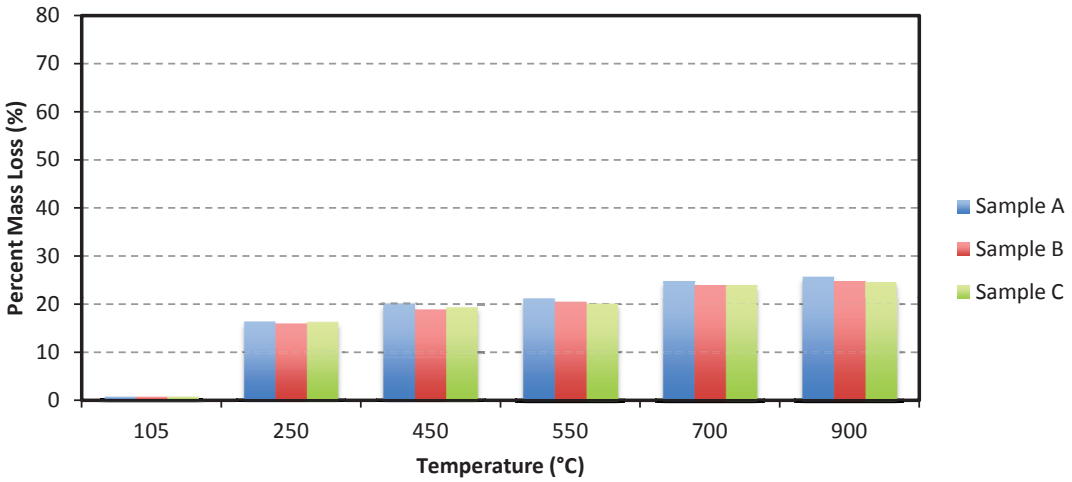
BH15A


80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

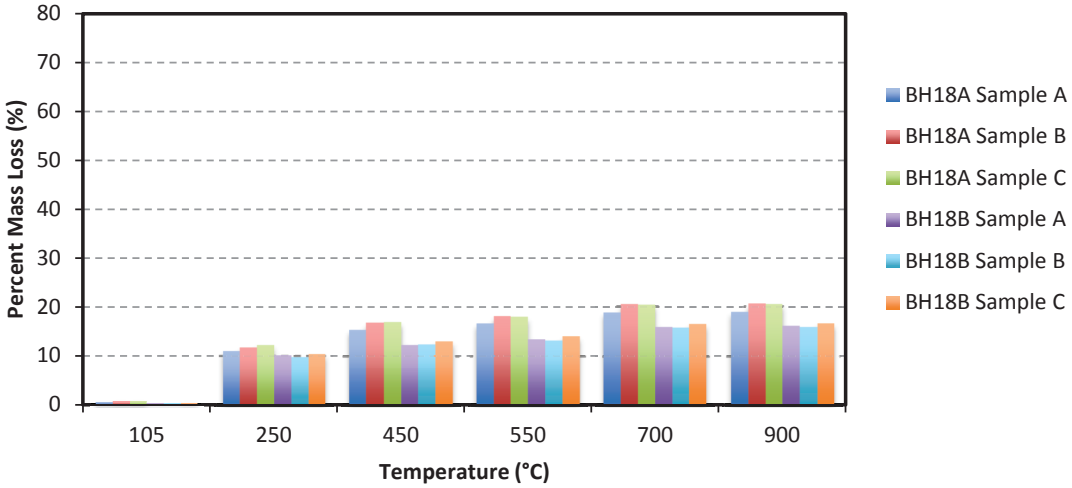
BH16A


80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH17A


80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

BH18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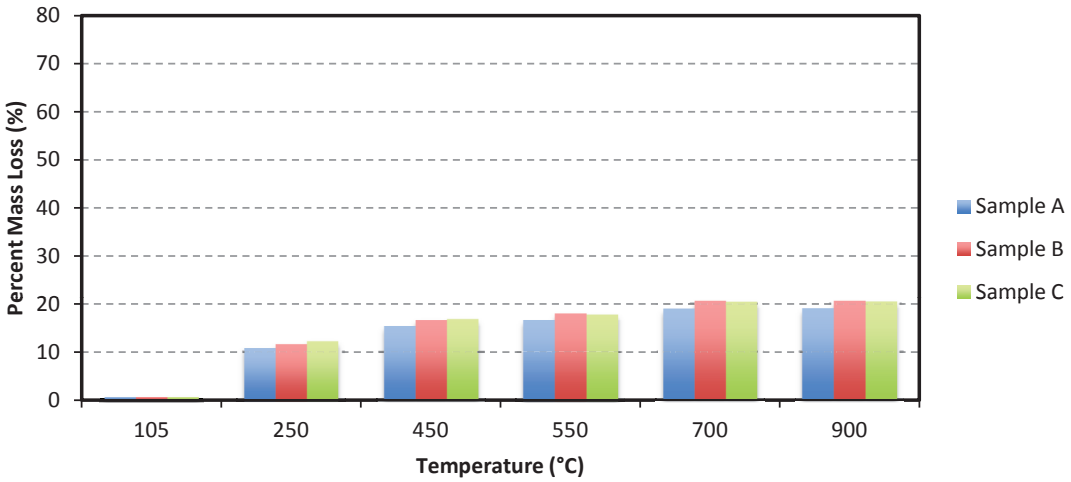
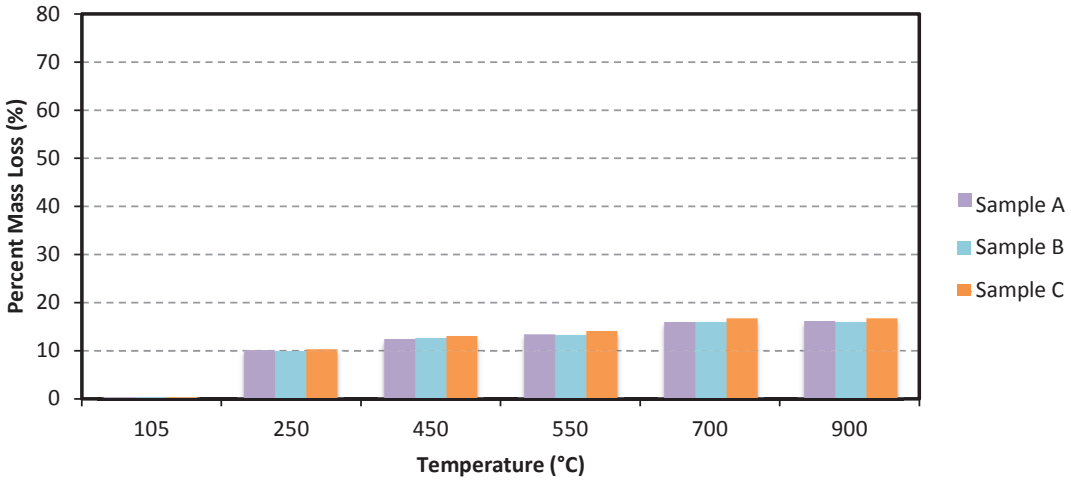
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
BH18A Sample A
BH18A Sample B
BH18A Sample C
BH18B Sample A
BH18B Sample B
BH18B Sample C

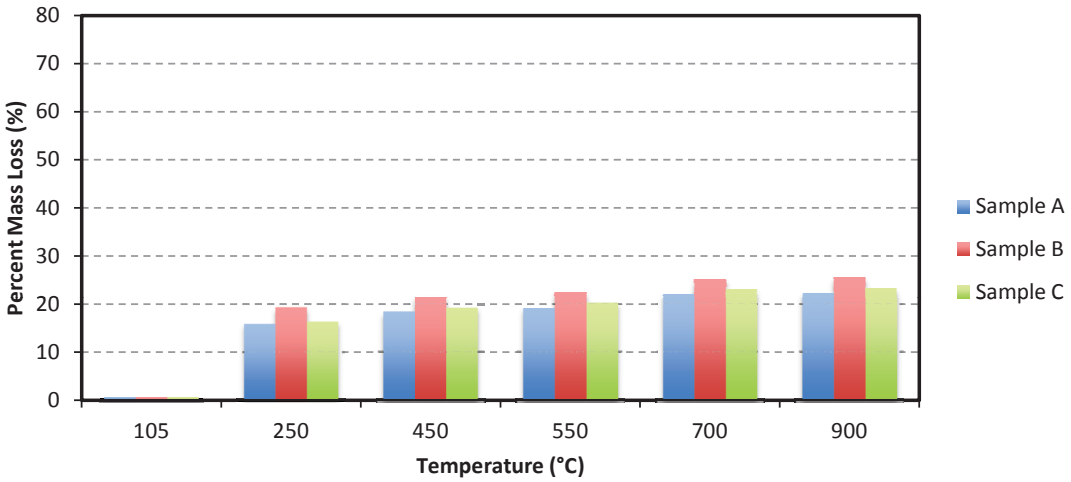
BH18A


80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

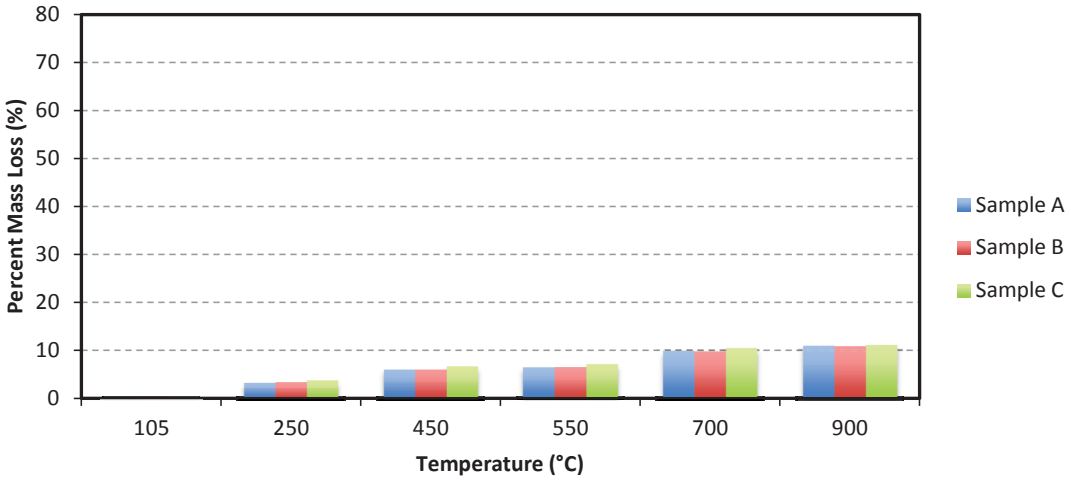
BH18B

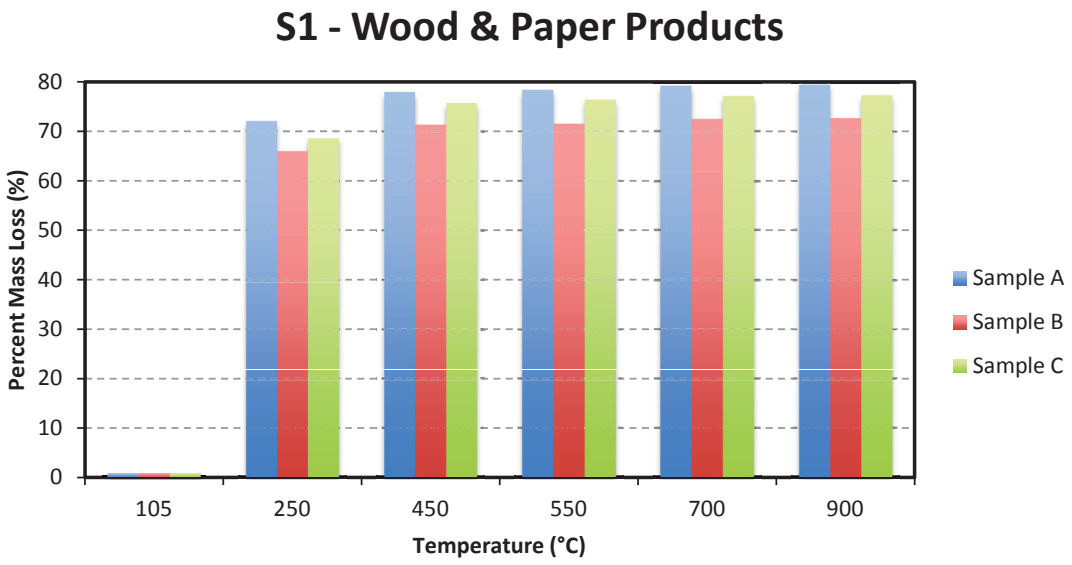

80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

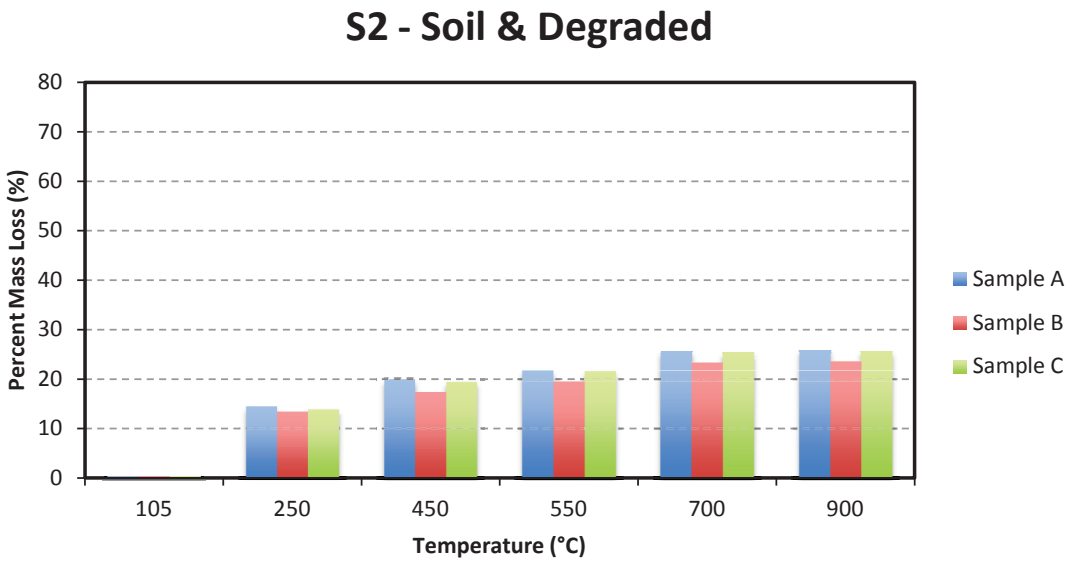
BH19A

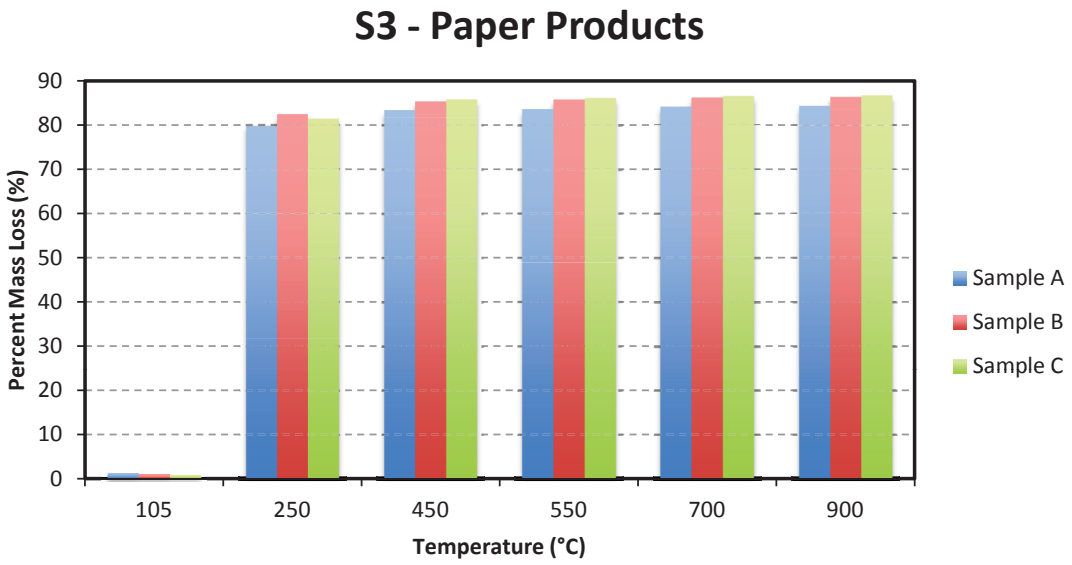

80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

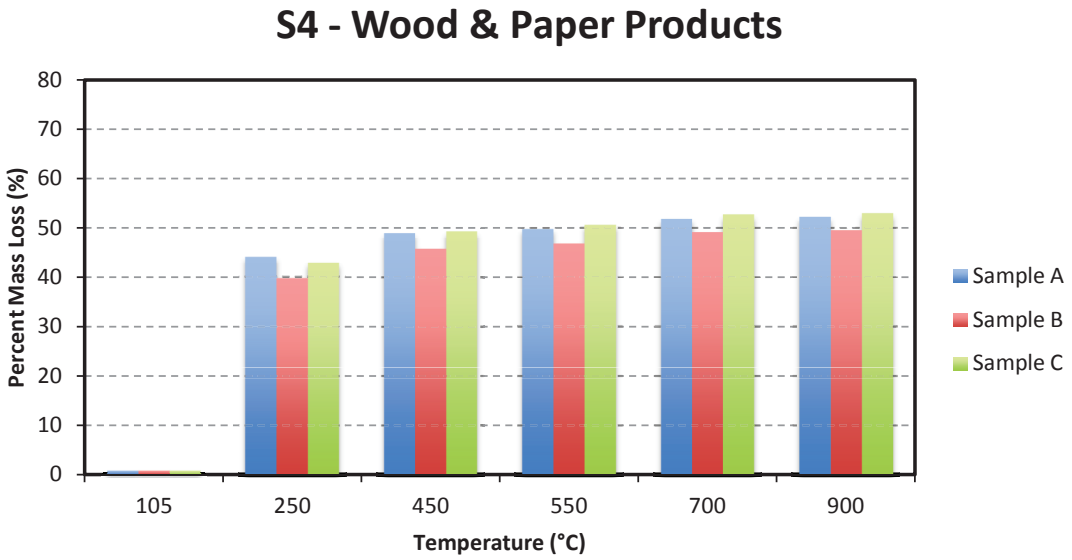
BH20A

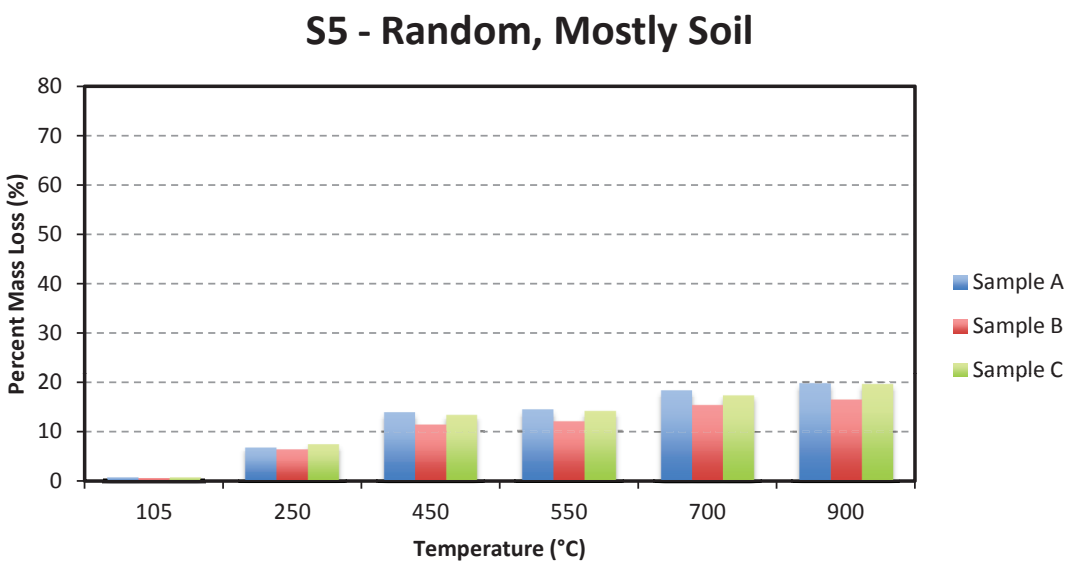

80
70
60
50
40
30
20
10
0
Percent Mass Loss (%)
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

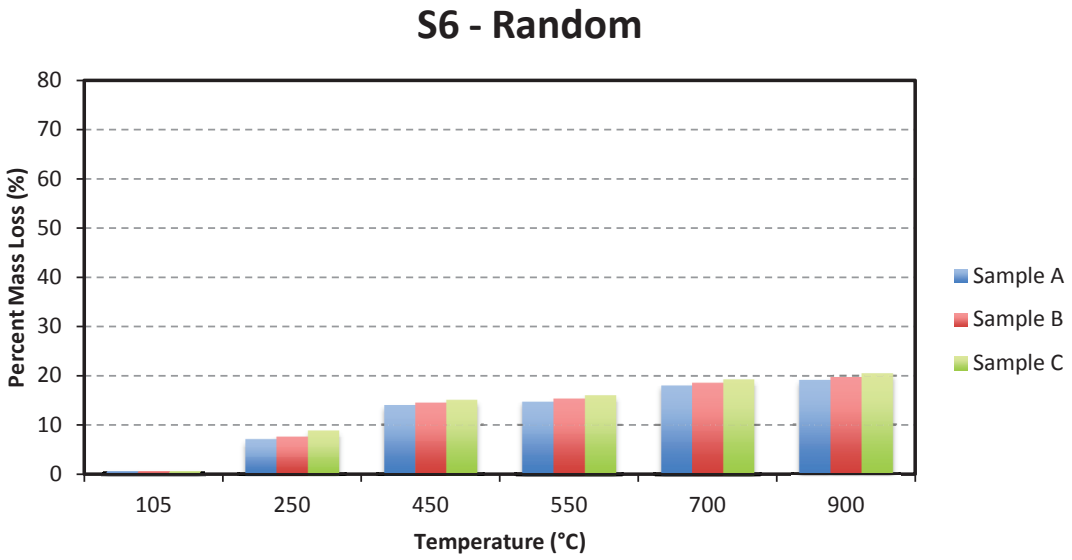

S1 - Wood & Paper Products
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

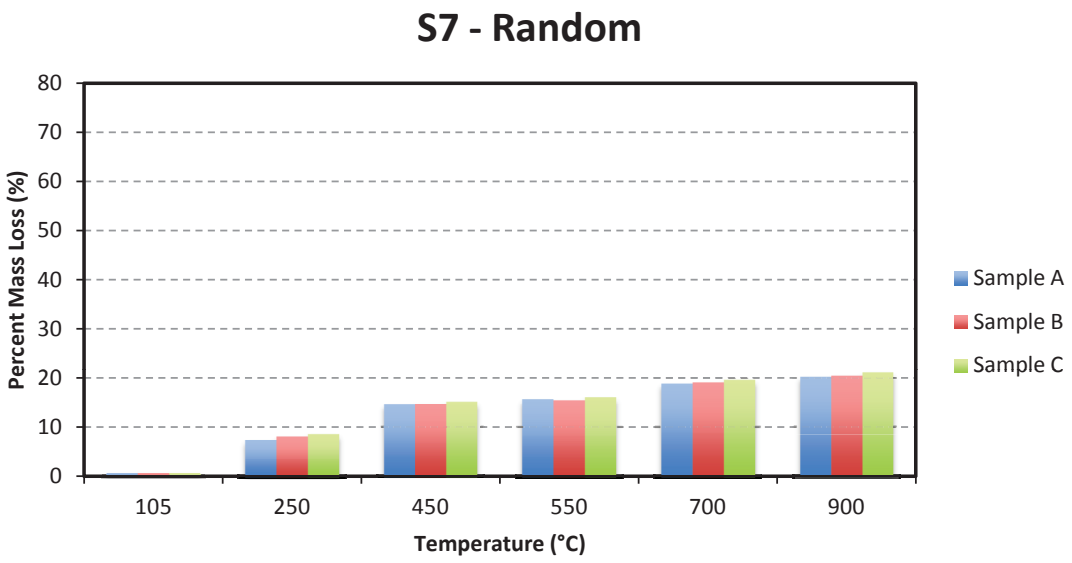

S2 - Soil & Degraded
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

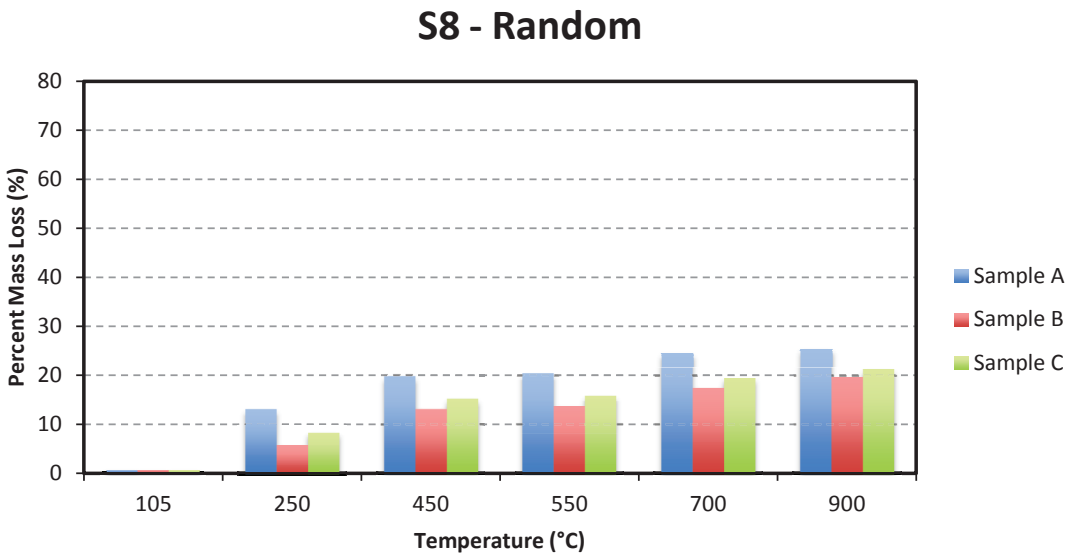

S3 - Paper Products
Percent Mass Loss (%)
90
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C


S4 - Wood & Paper Products
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

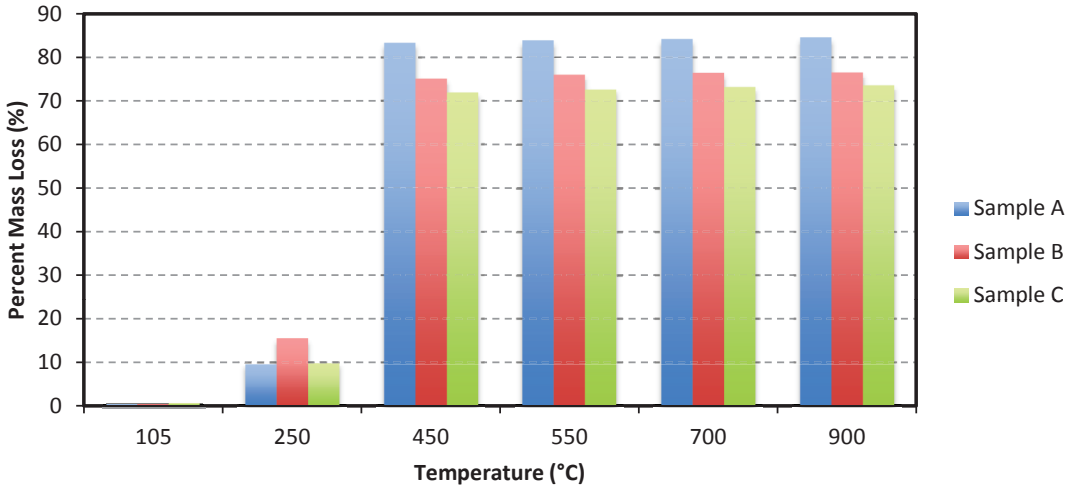

S5 - Random, Mostly Soil
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C


S6 - Random
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

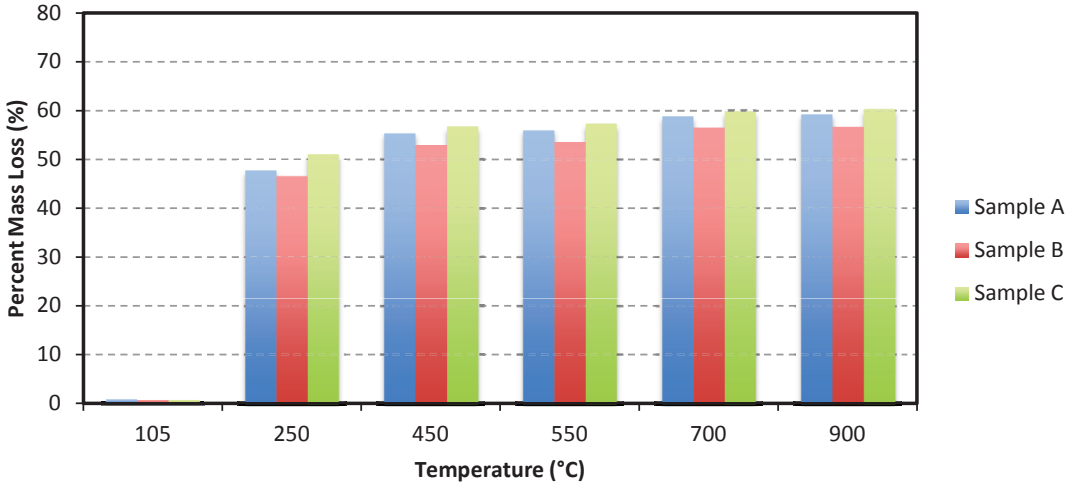

S7 - Random
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

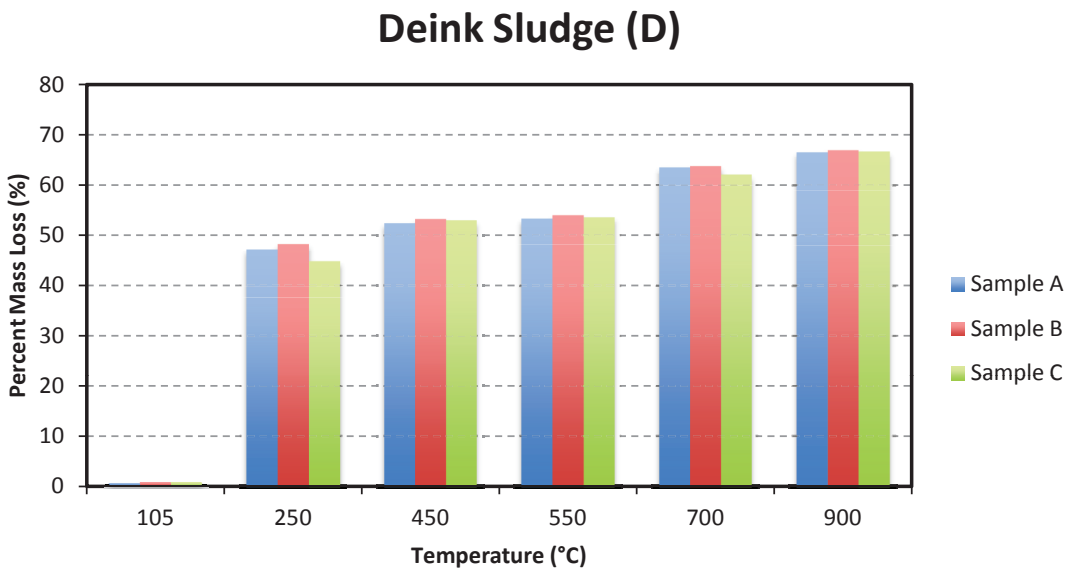

S8 - Random
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

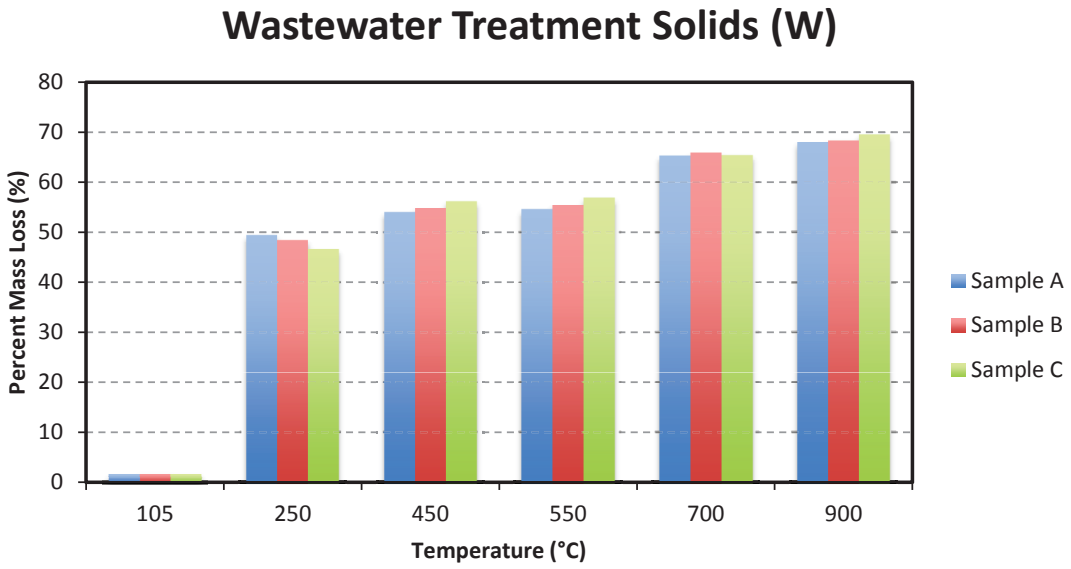
S9 - Plastics

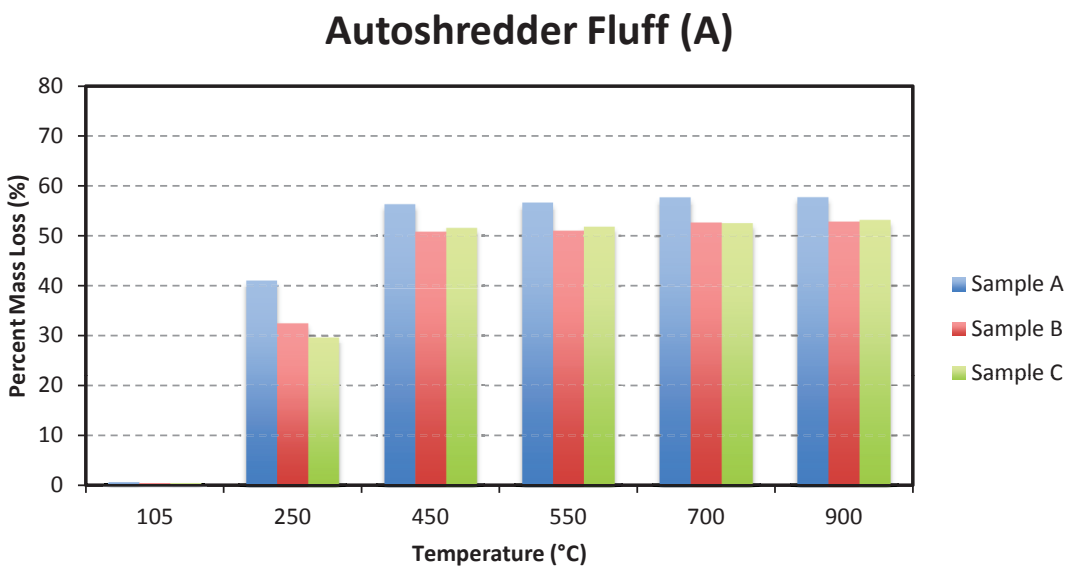


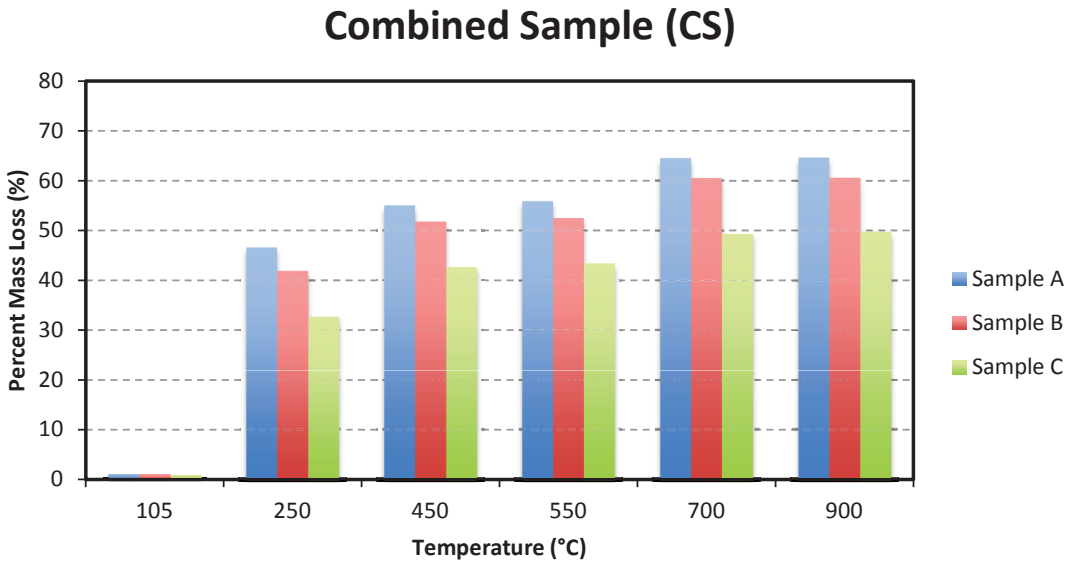
S10 - Wood

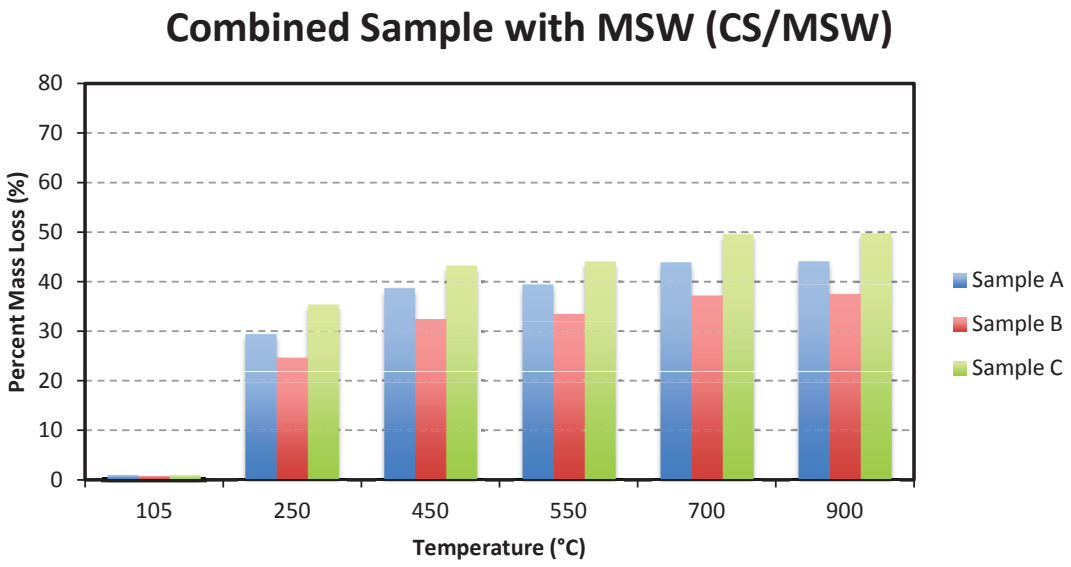



Deink Sludge (D)
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C


Wastewater Treatment Solids (W)
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

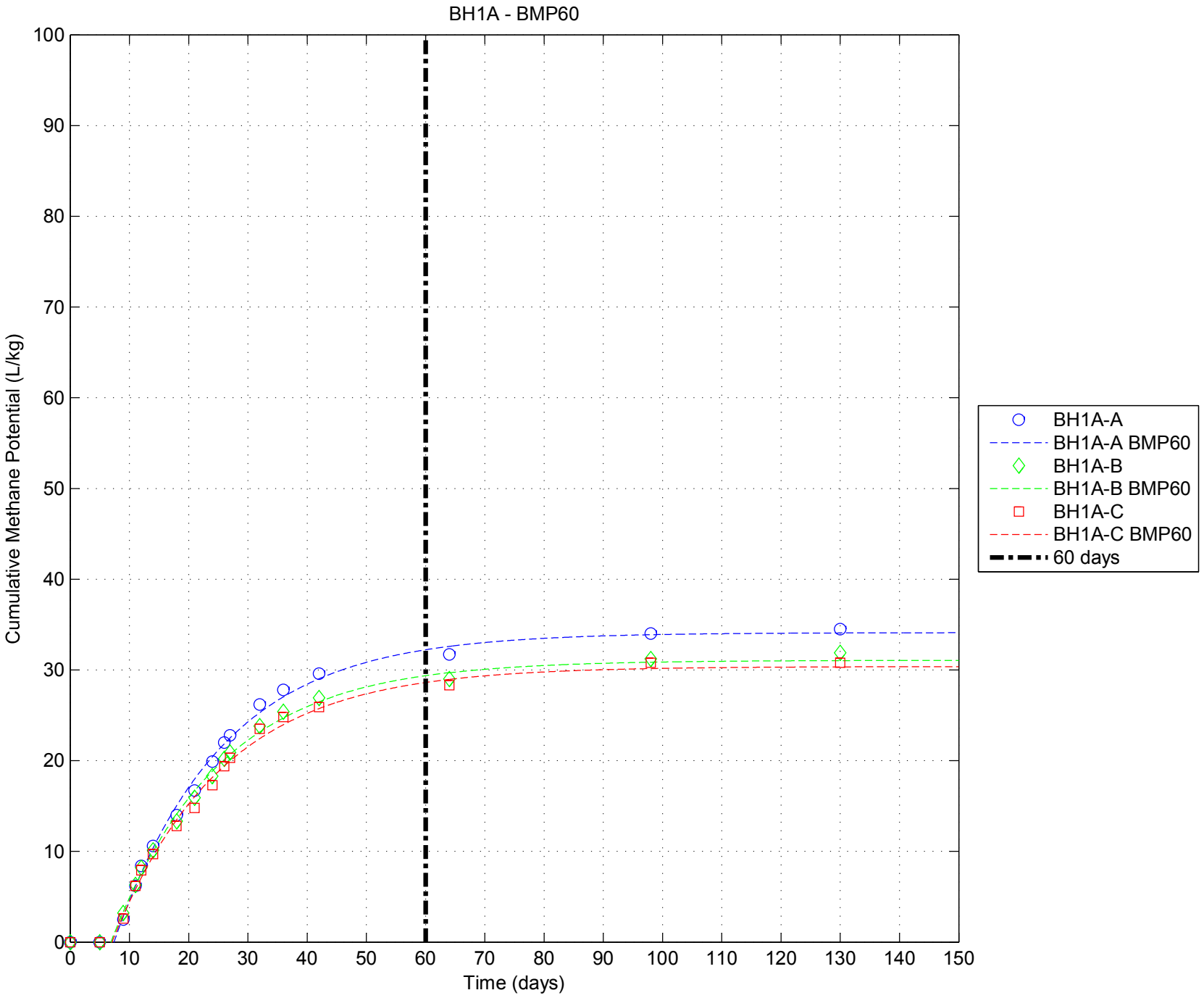

Autoshredder Fluff (A)
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

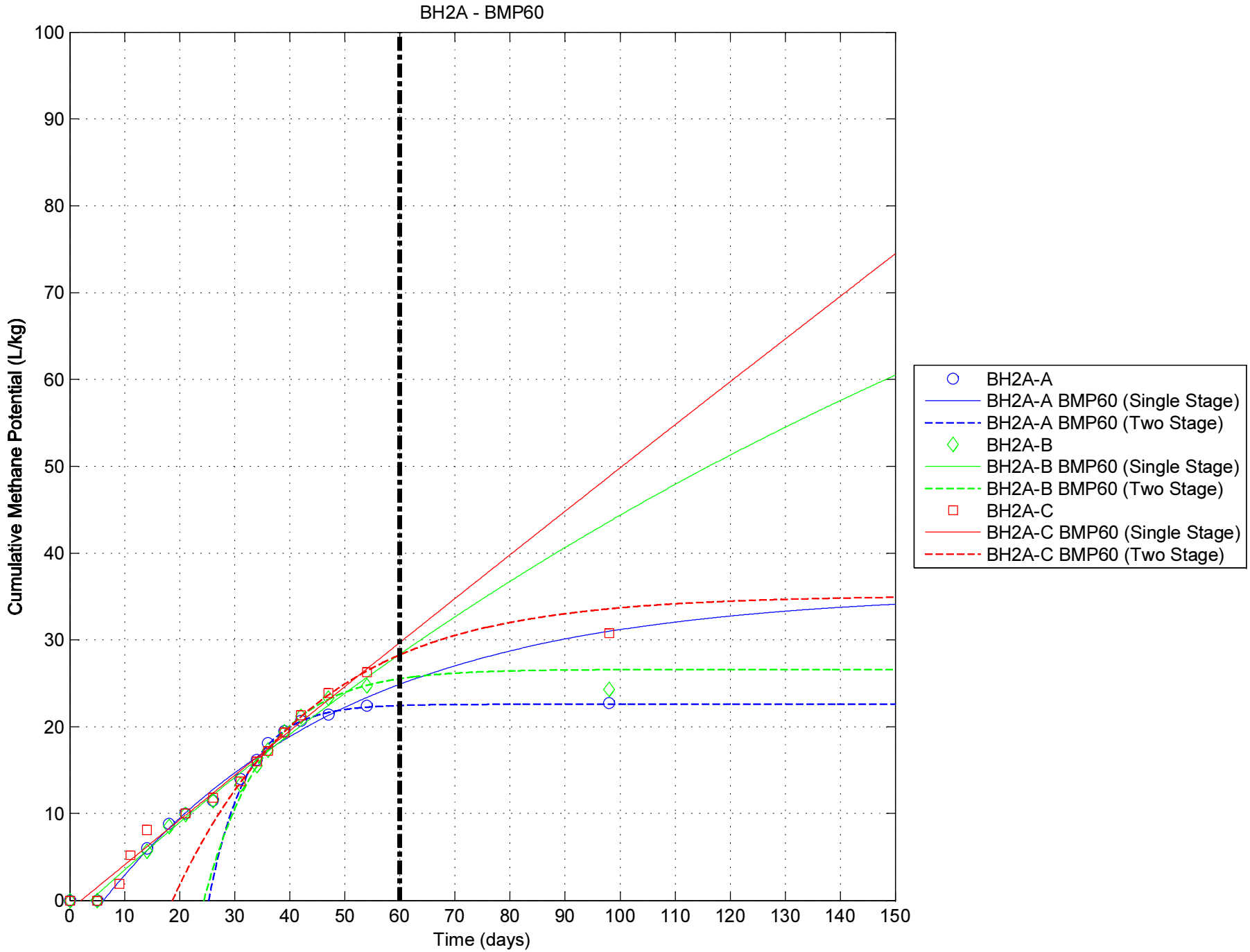

Combined Sample (CS)
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

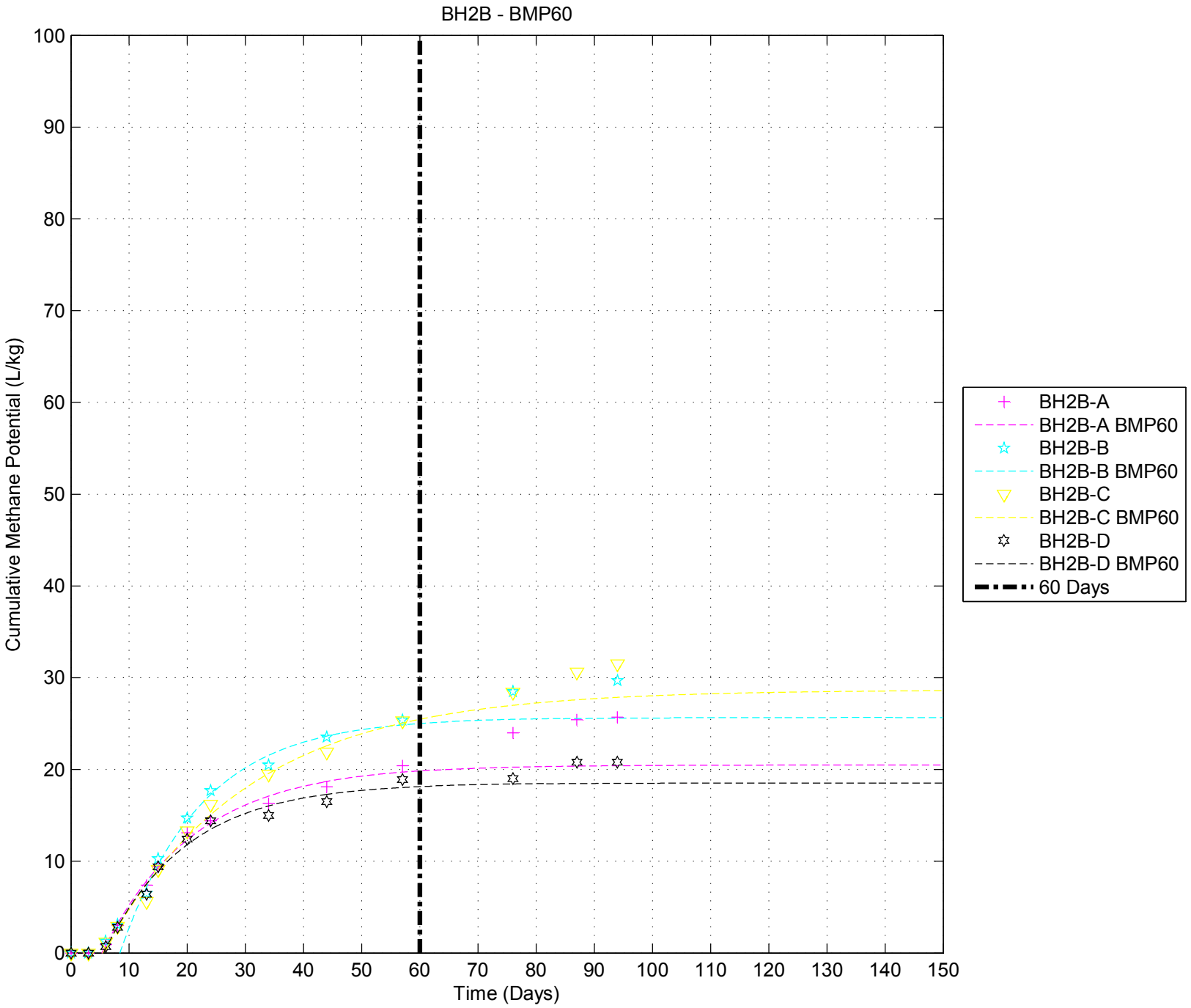

Combined Sample with MSW (CS/MSW)
Percent Mass Loss (%)
80
70
60
50
40
30
20
10
0
105
250
450
550
700
900
Temperature (°C)
Sample A
Sample B
Sample C

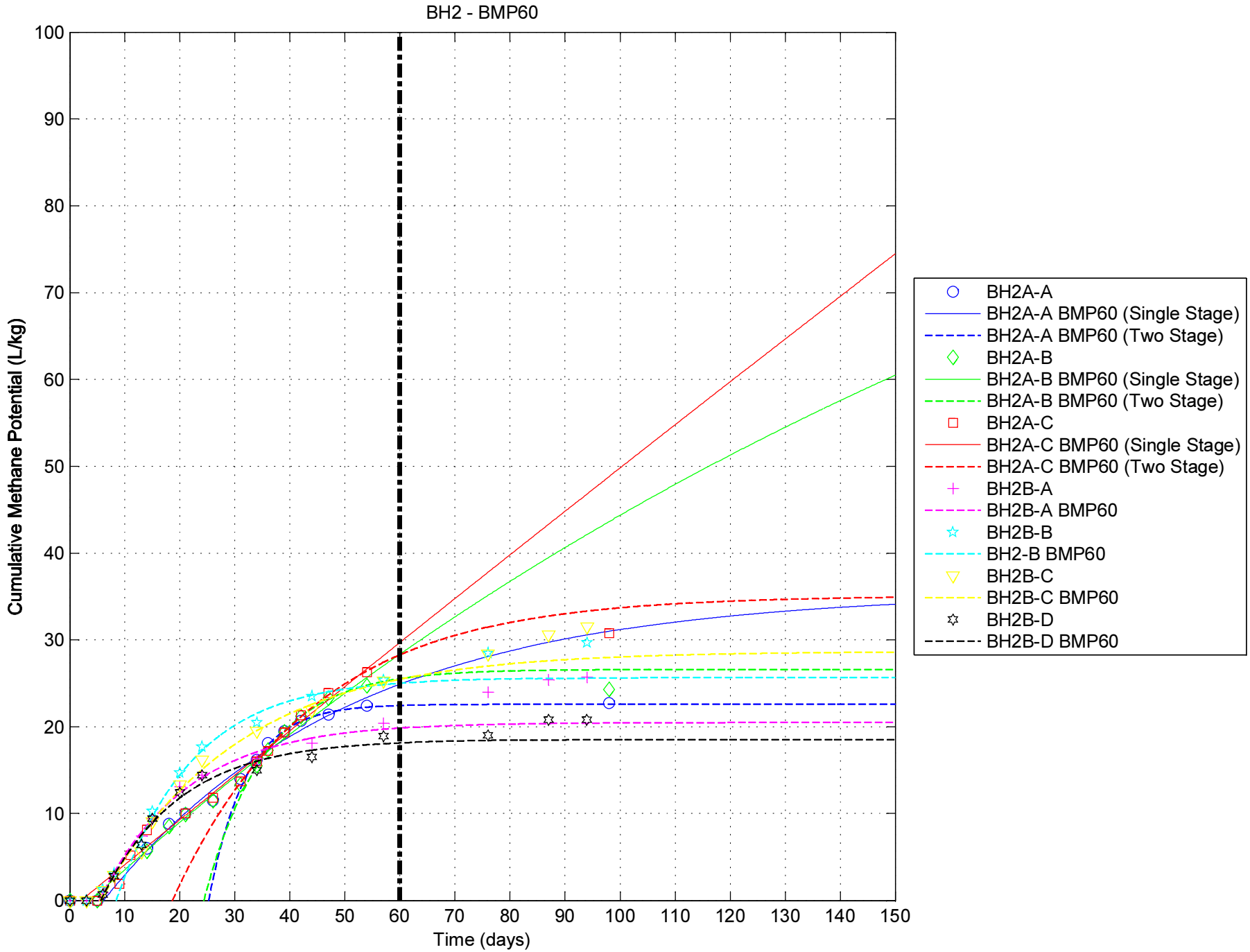
Appendix E

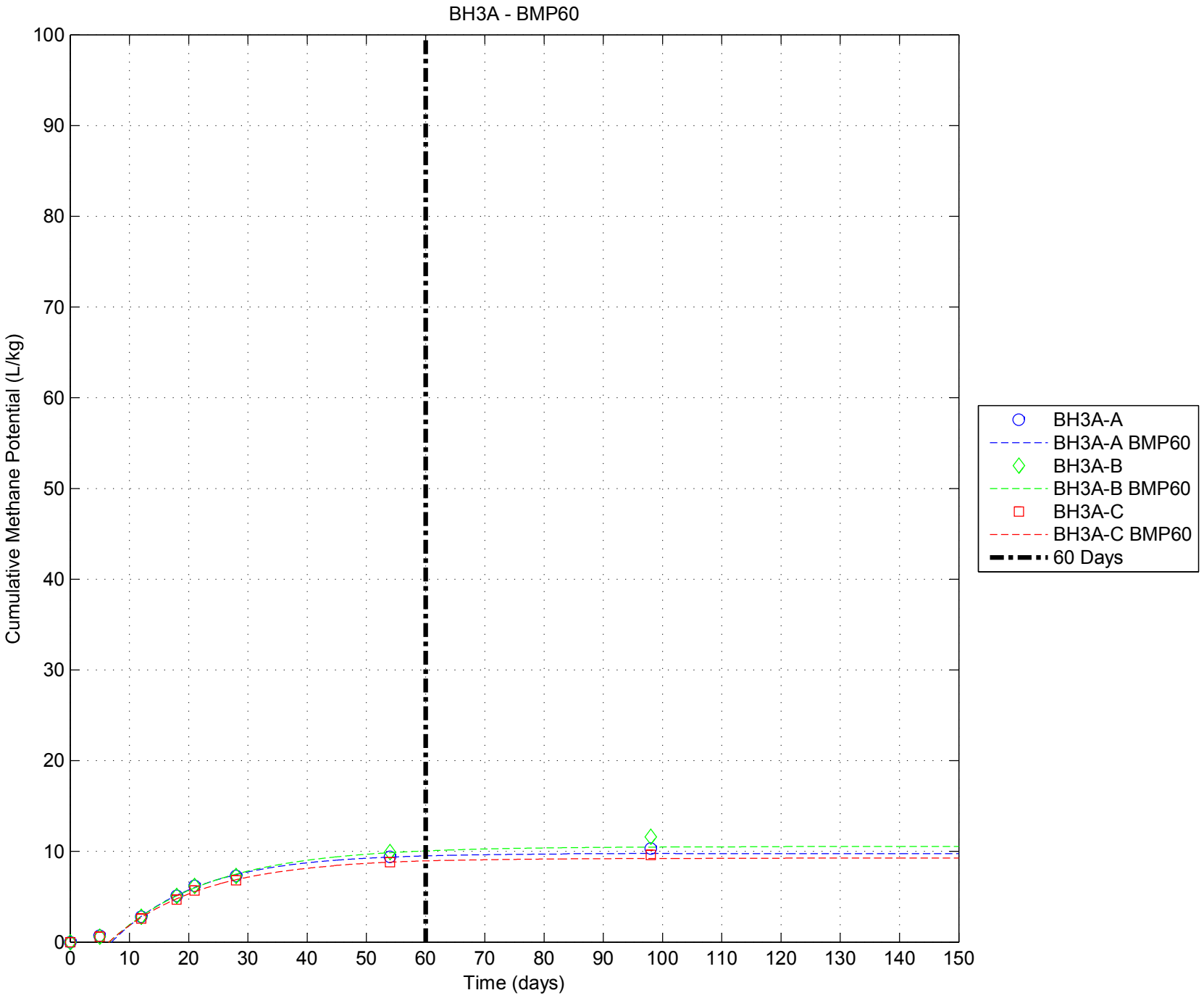
BMP60 Plots

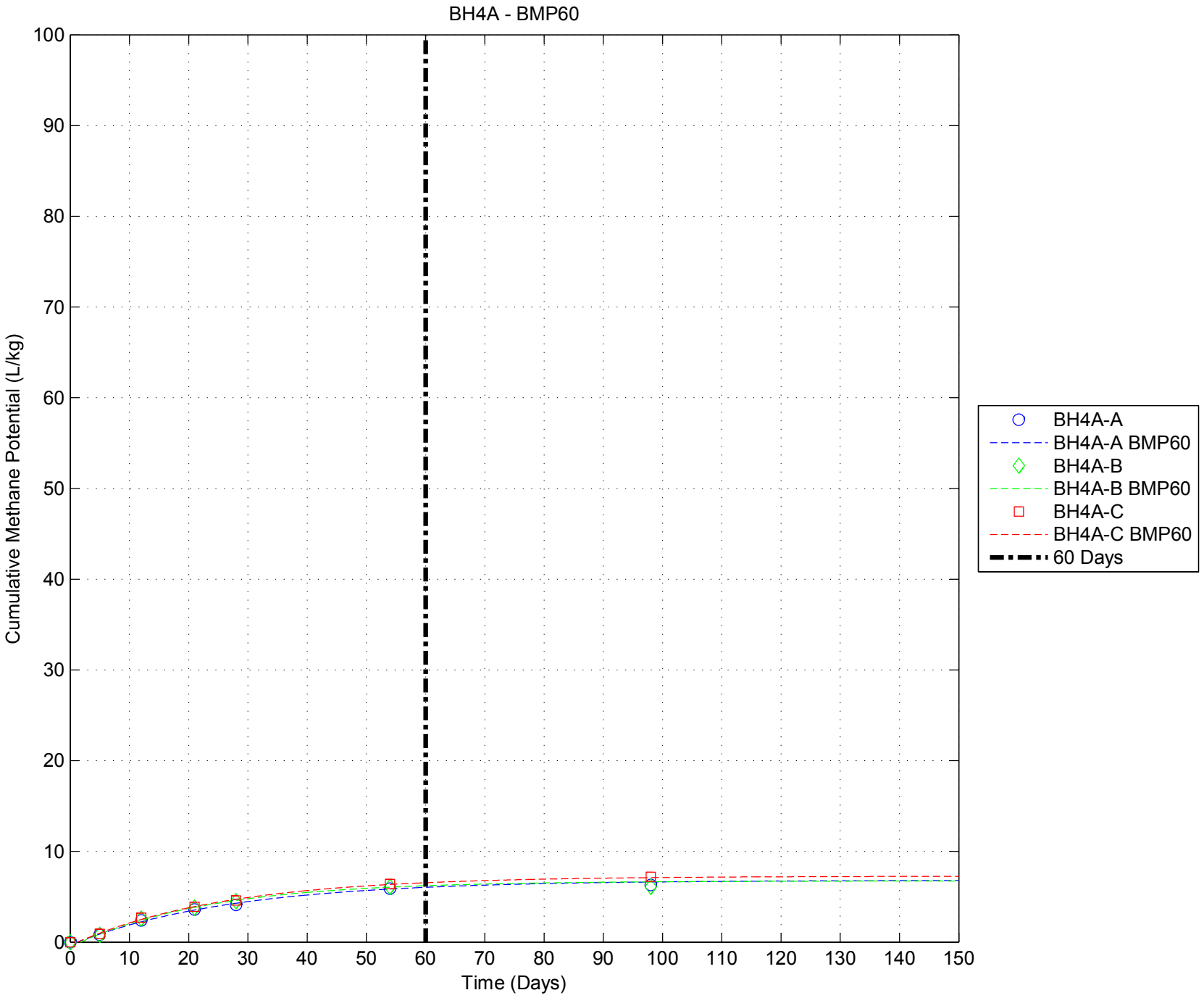

BH1A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH1A-A
BH1A-A BMP60
BH1A-B
BH1A-B BMP60
BH1A-C
BH1A-C BMP60
60 days

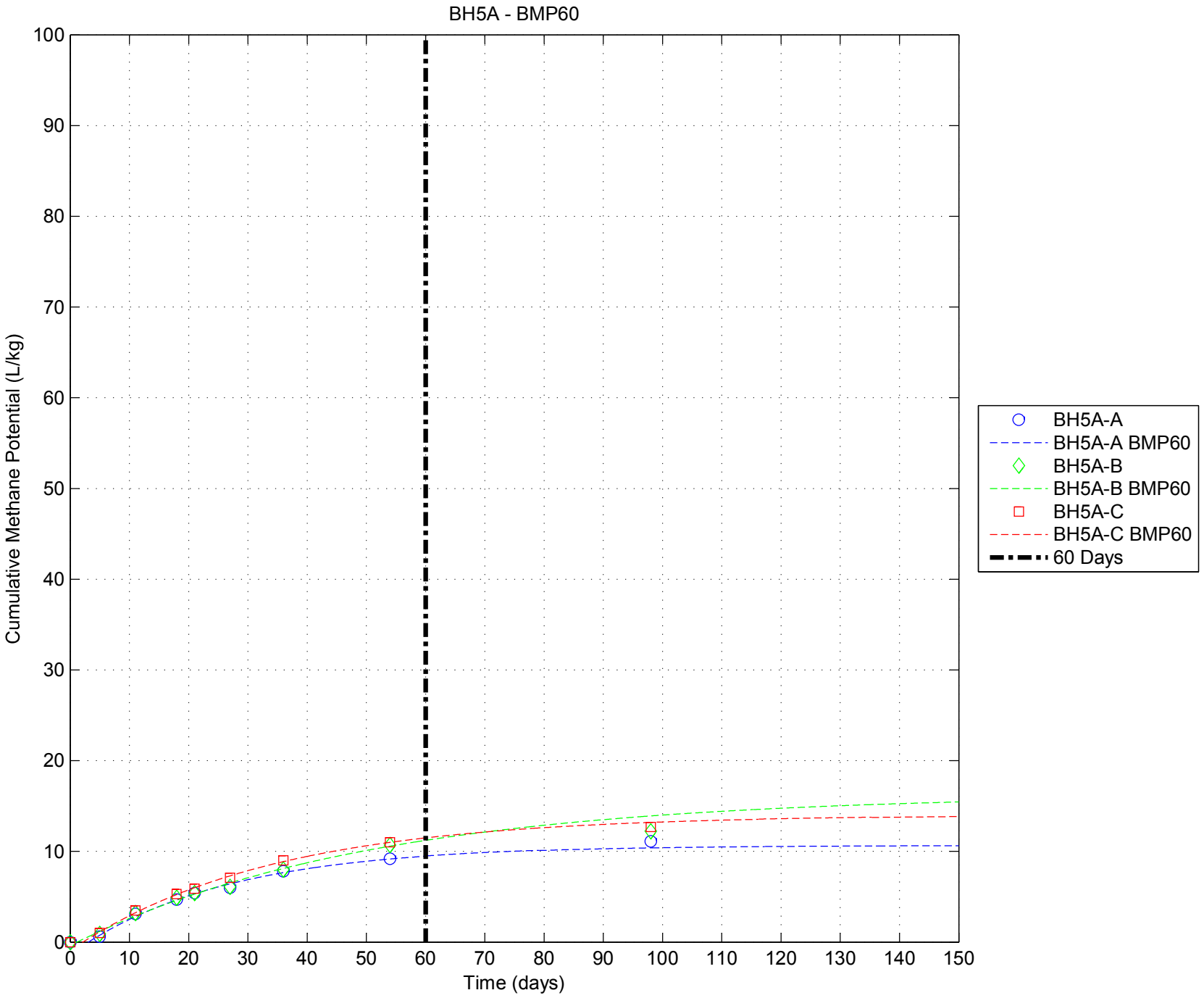

BH2A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH2A-A
BH2A-A BMP60 (Single Stage)
BH2A-A BMP60 (Two Stage)
BH2A-B
BH2A-B BMP60 (Single Stage)
BH2A-B BMP60 (Two Stage)
BH2A-C
BH2A-C BMP60 (Single Stage)
BH2A-C BMP60 (Two Stage)

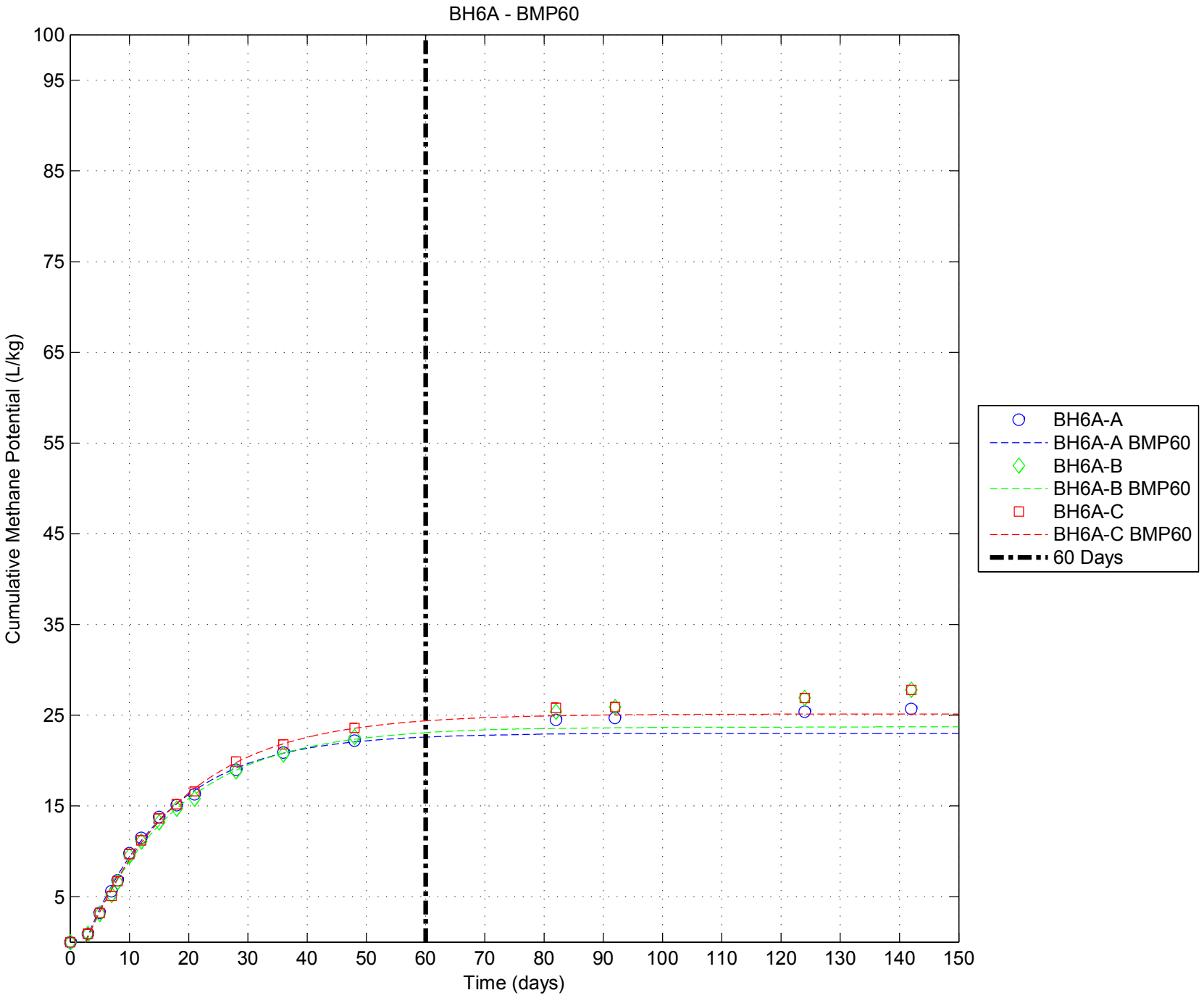

BH2B - BMP60
Cumulative Methane Potential (L/kg)
Time (Days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH2B-A
BH2B-A BMP60
BH2B-B
BH2B-B BMP60
BH2B-C
BH2B-C BMP60
BH2B-D
BH2B-D BMP60
60 Days

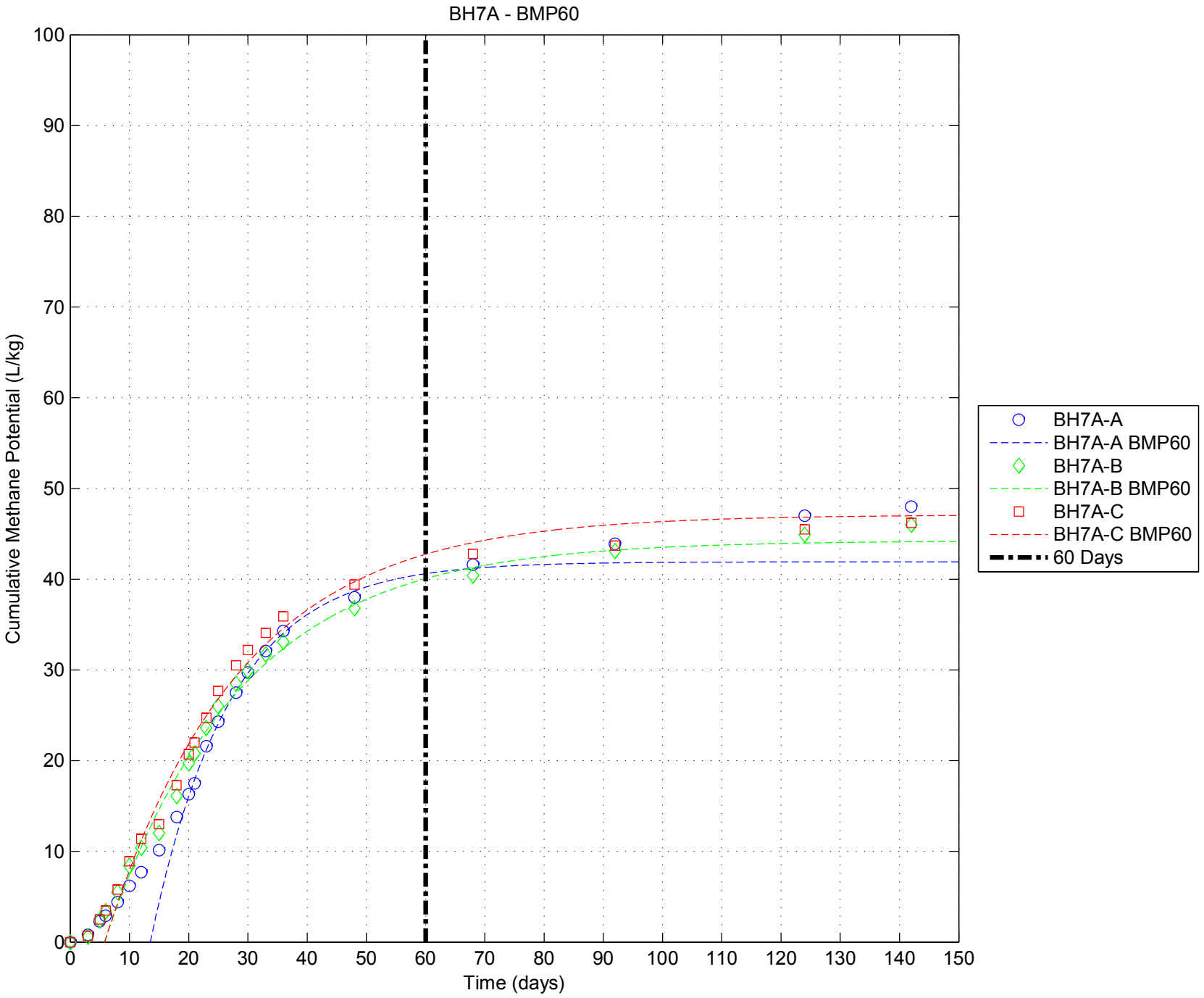

BH2 - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH2A-A
BH2A-A BMP60 (Single Stage)
BH2A-A BMP60 (Two Stage)
BH2A-B
BH2A-B BMP60 (Single Stage)
BH2A-B BMP60 (Two Stage)
BH2A-C
BH2A-C BMP60 (Single Stage)
BH2A-C BMP60 (Two Stage)
BH2B-A
BH2B-A BMP60
BH2B-B
BH2-B BMP60
BH2B-C
BH2B-C BMP60
BH2B-D
BH2B-D BMP60

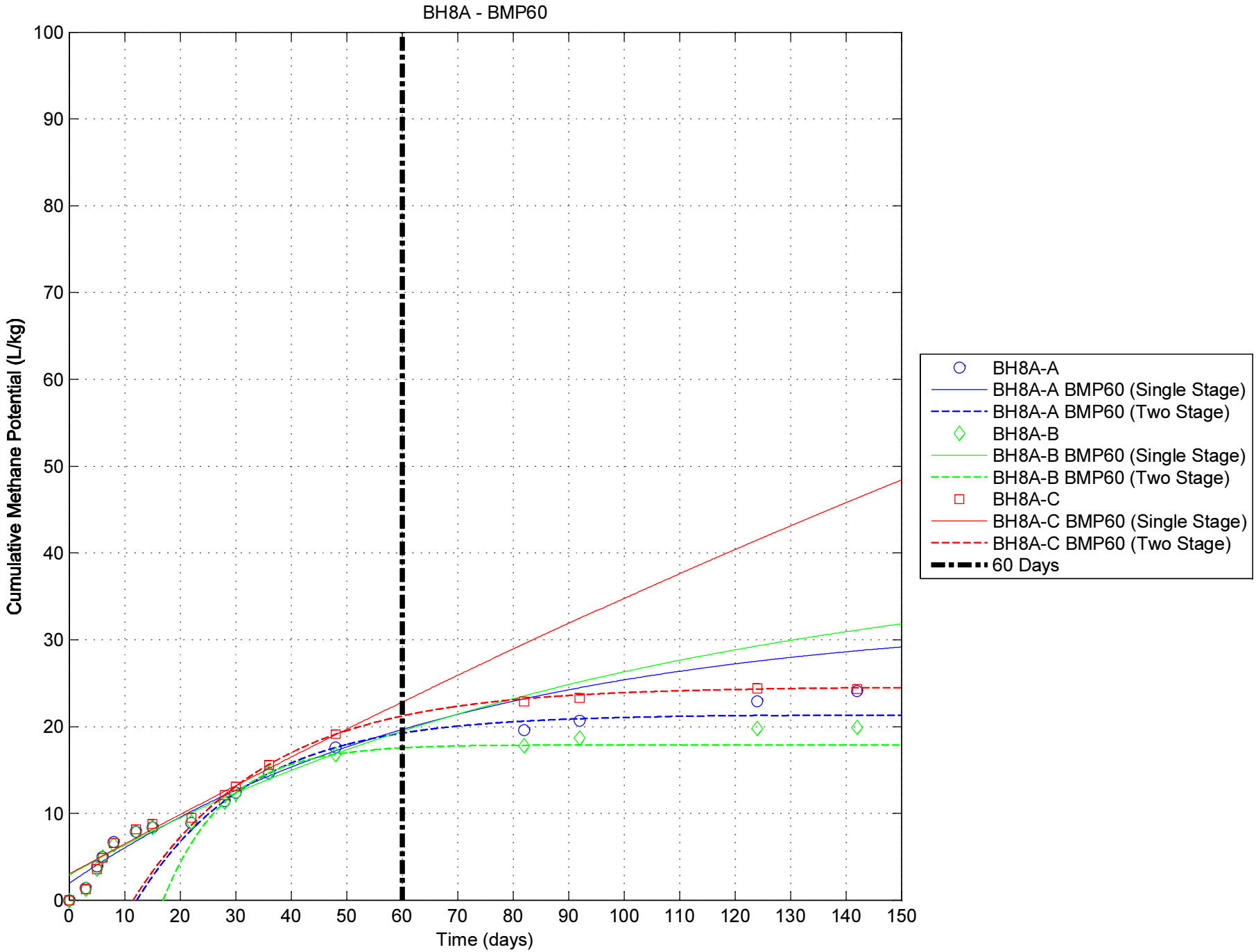

BH3A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH3A-A
BH3A-A BMP60
BH3A-B
BH3A-B BMP60
BH3A-C
BH3A-C BMP60
60 Days

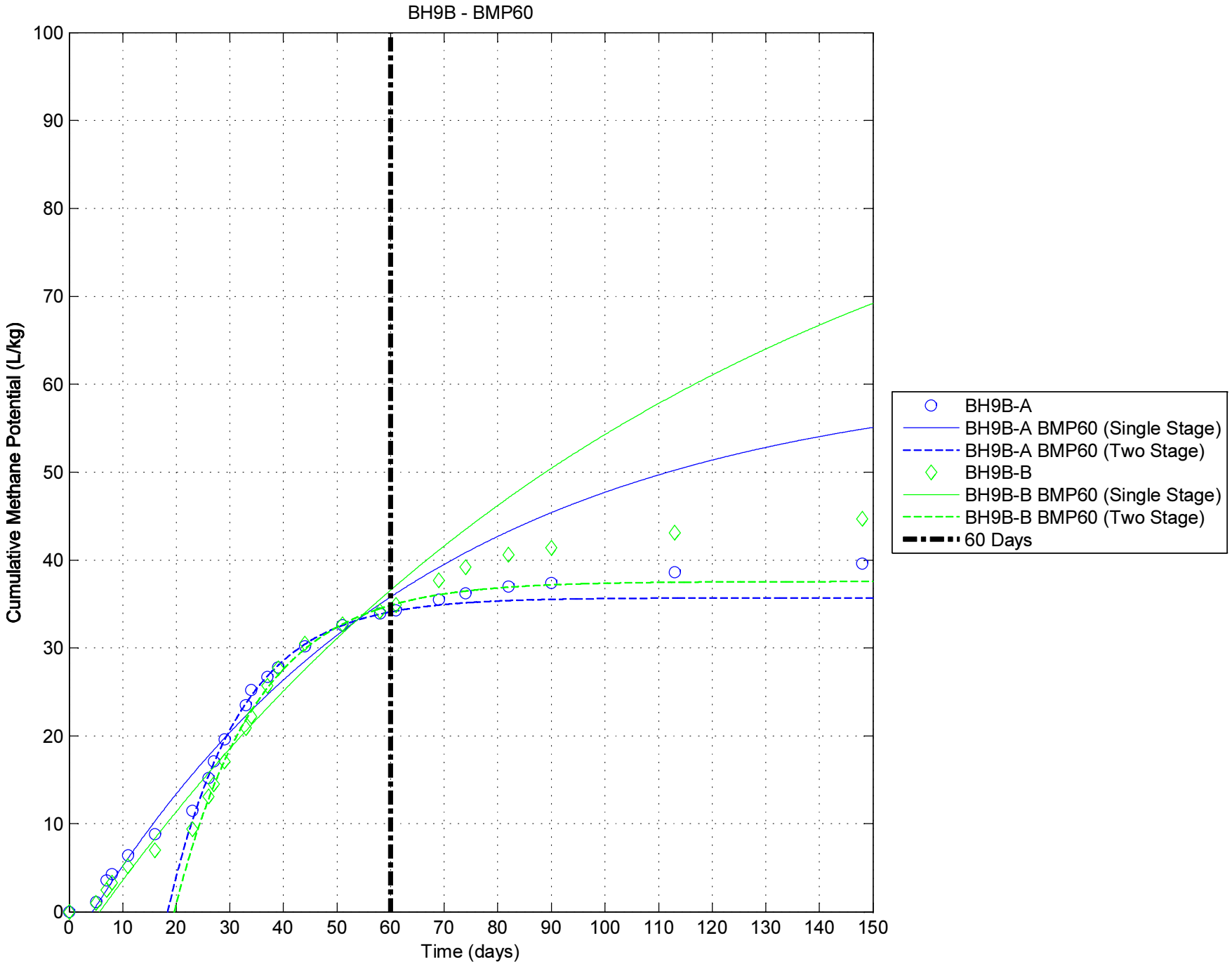

BH4A - BMP60
Cumulative Methane Potential (L/kg)
Time (Days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH4A-A
BH4A-A BMP60
BH4A-B
BH4A-B BMP60
BH4A-C
BH4A-C BMP60
60 Days

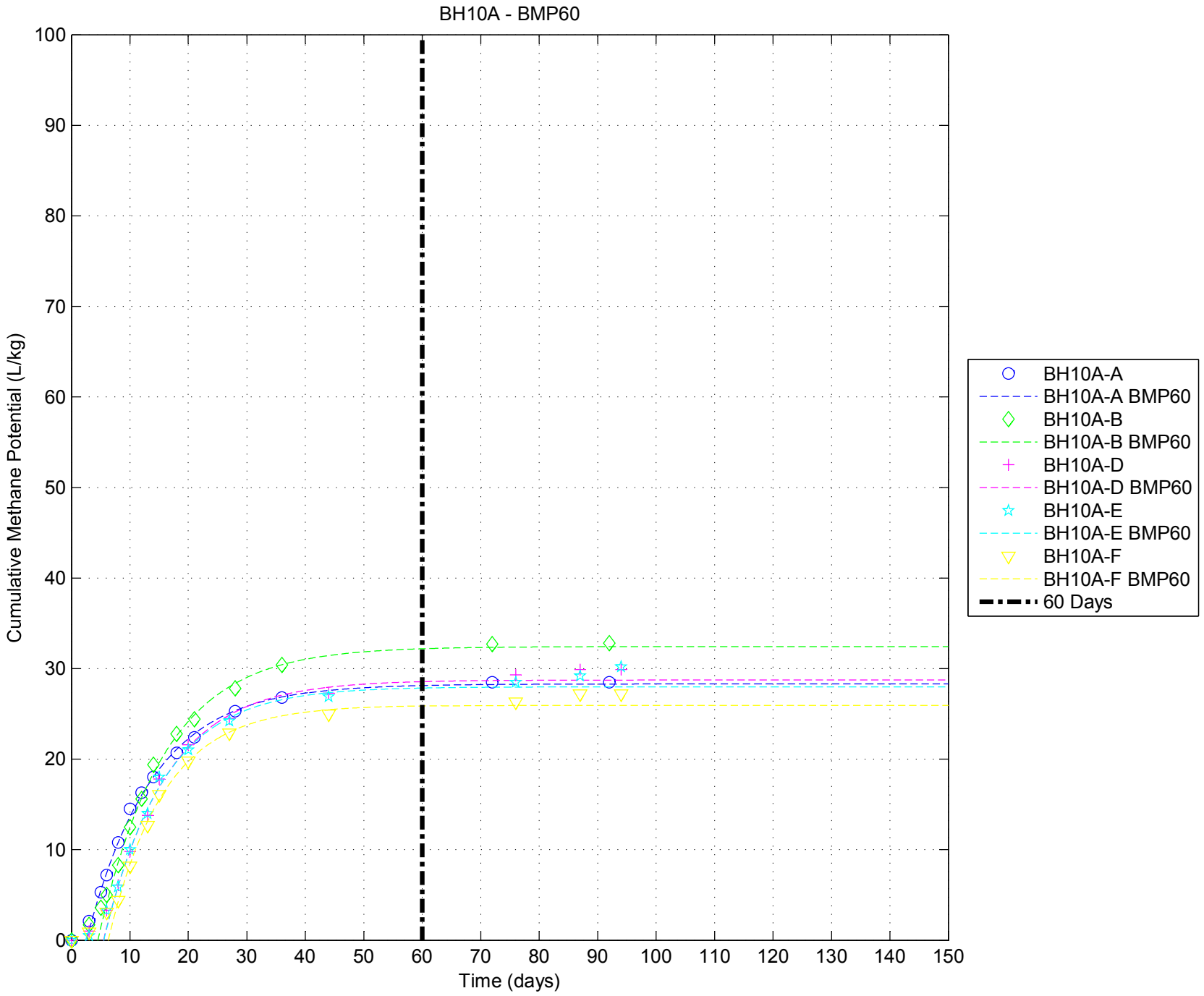

BH5A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH5A-A
BH5A-A BMP60
BH5A-B
BH5A-B BMP60
BH5A-C
BH5A-C BMP60
60 Days

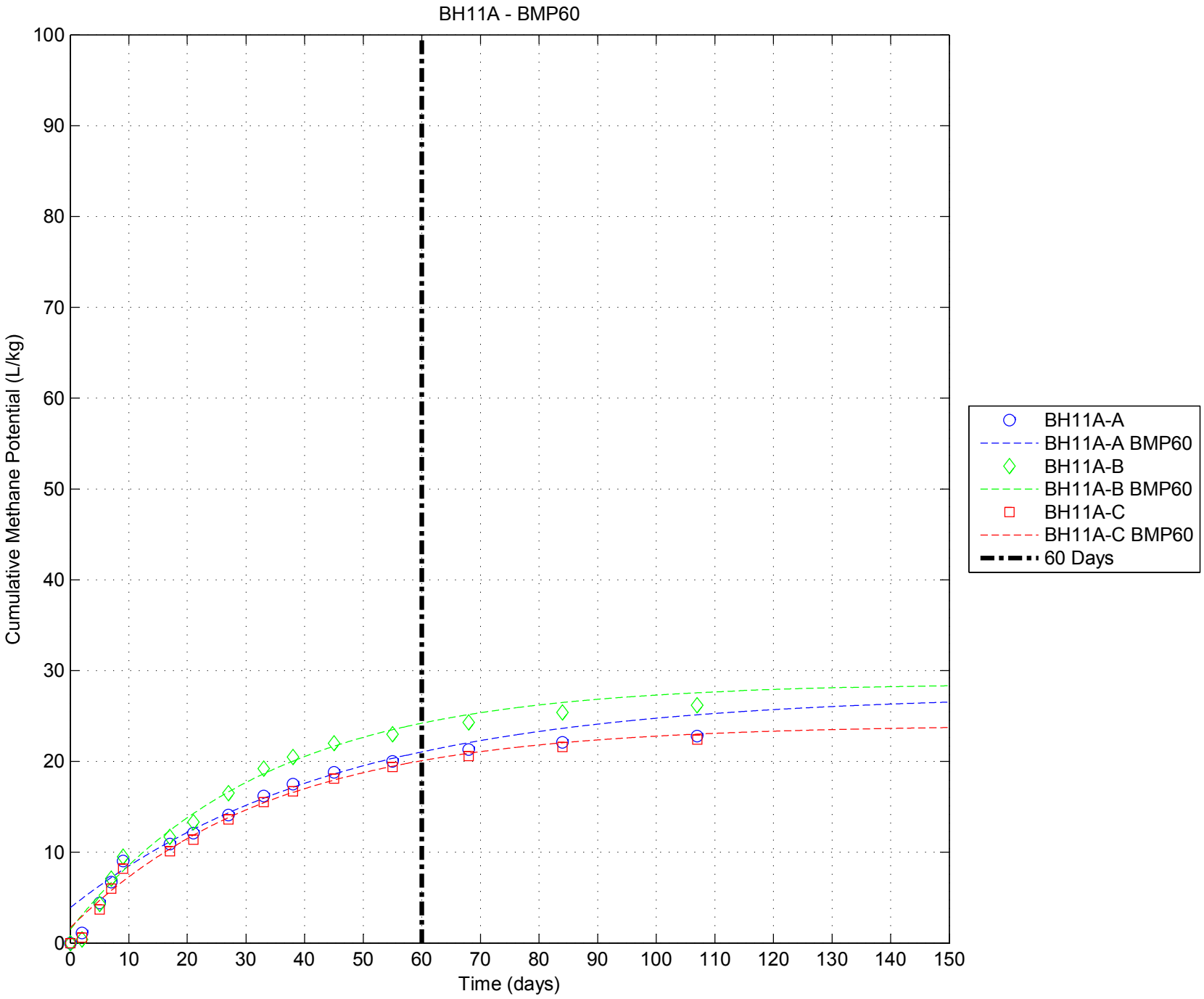

BH6A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
BH6A-A
BH6A-A BMP60
BH6A-B
BH6A-B BMP60
BH6A-C
BH6A-C BMP60
60 Days

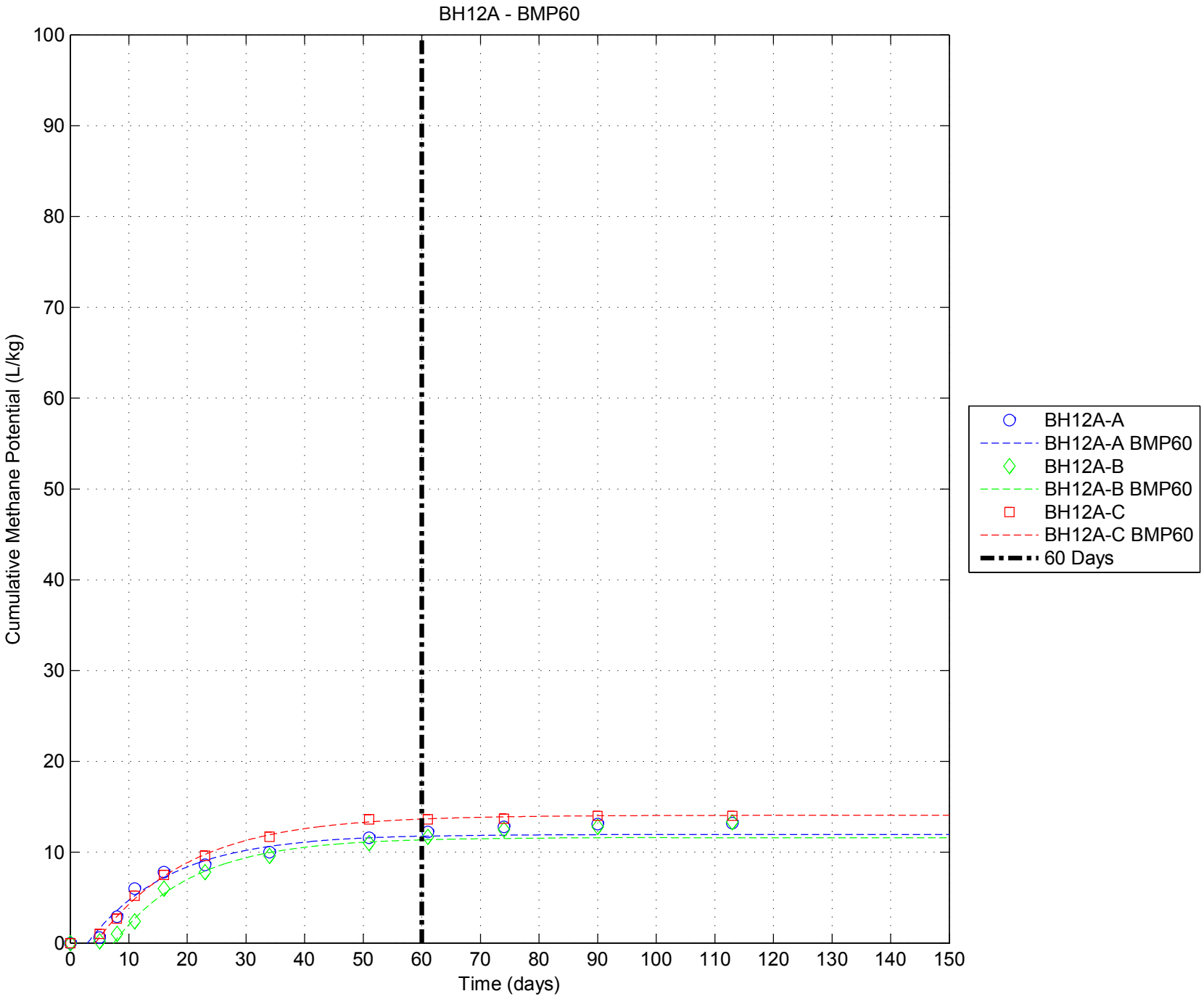

BH7A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH7A-A
BH7A-A BMP60
BH7A-B
BH7A-B BMP60
BH7A-C
BH7A-C BMP60
60 Days

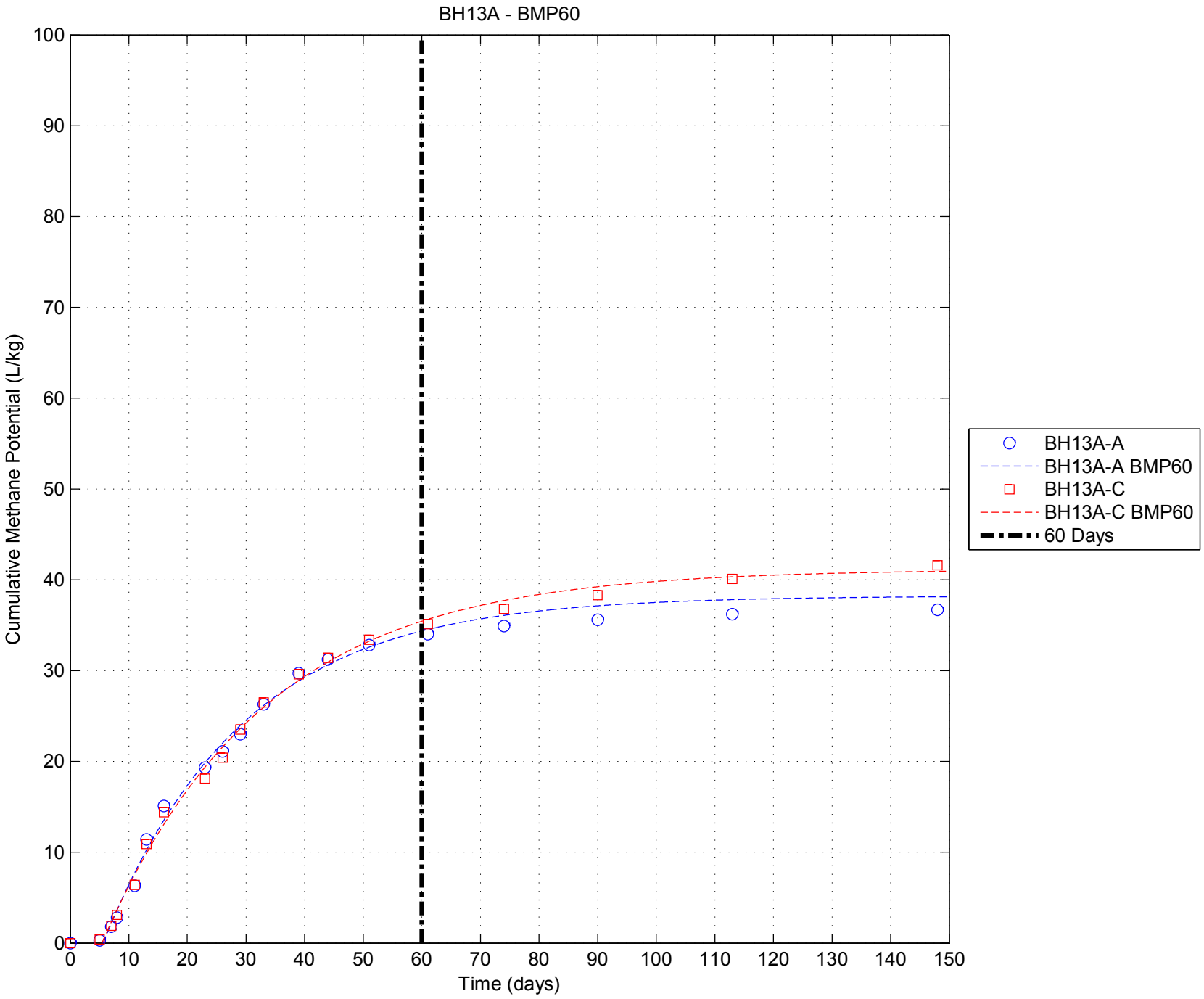

BH8A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH8A-A
BH8A-A BMP60 (Single Stage)
BH8A-A BMP60 (Two Stage)
BH8A-B
BH8A-B BMP60 (Single Stage)
BH8A-B BMP60 (Two Stage)
BH8A-C
BH8A-C BMP60 (Single Stage)
BH8A-C BMP60 (Two Stage)
60 Days

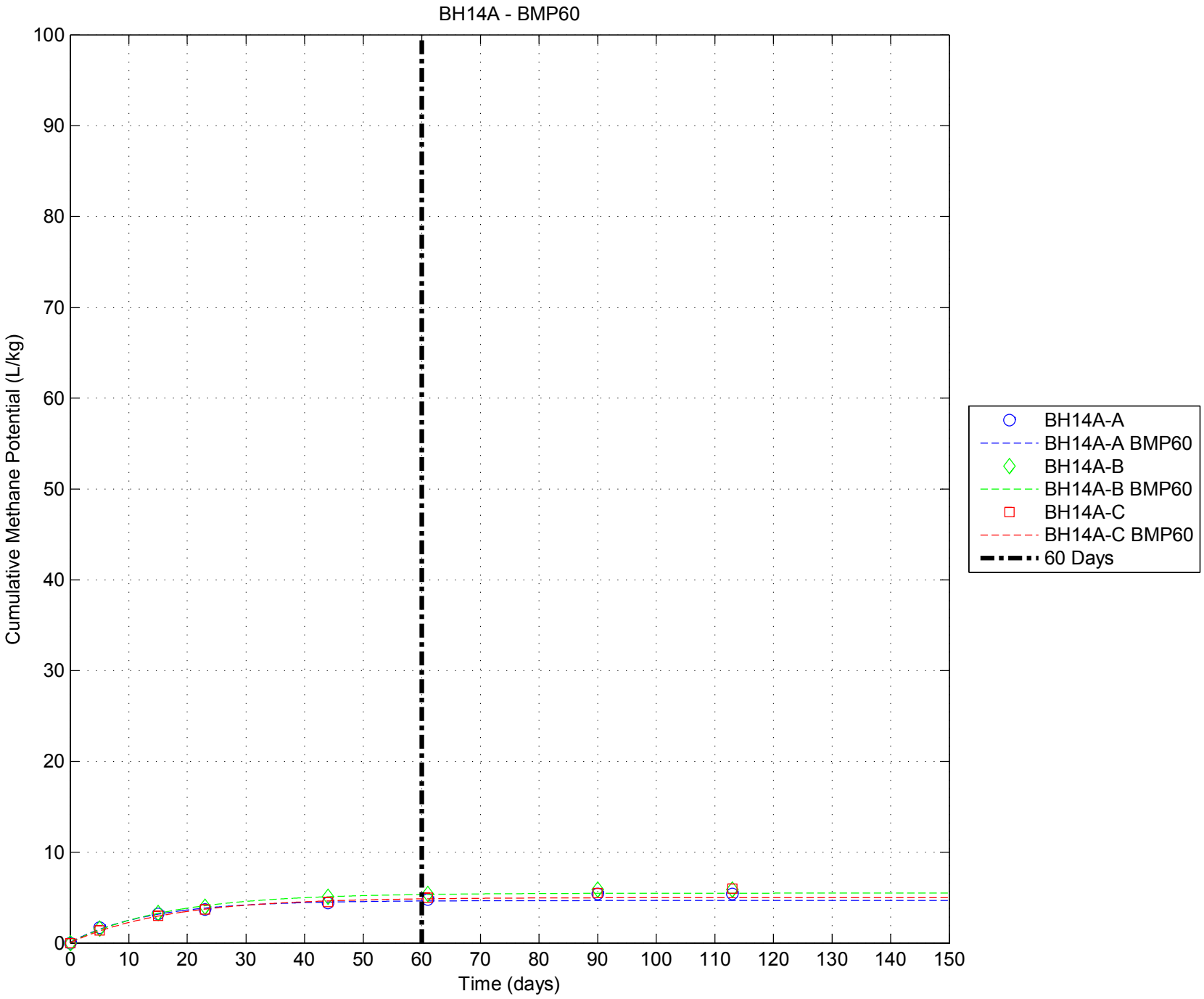

BH9B - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH9B-A
BH9B-A BMP60 (Single Stage)
BH9B-A BMP60 (Two Stage)
BH9B-B
BH9B-B BMP60 (Single Stage)
BH9B-B BMP60 (Two Stage)
60 Days

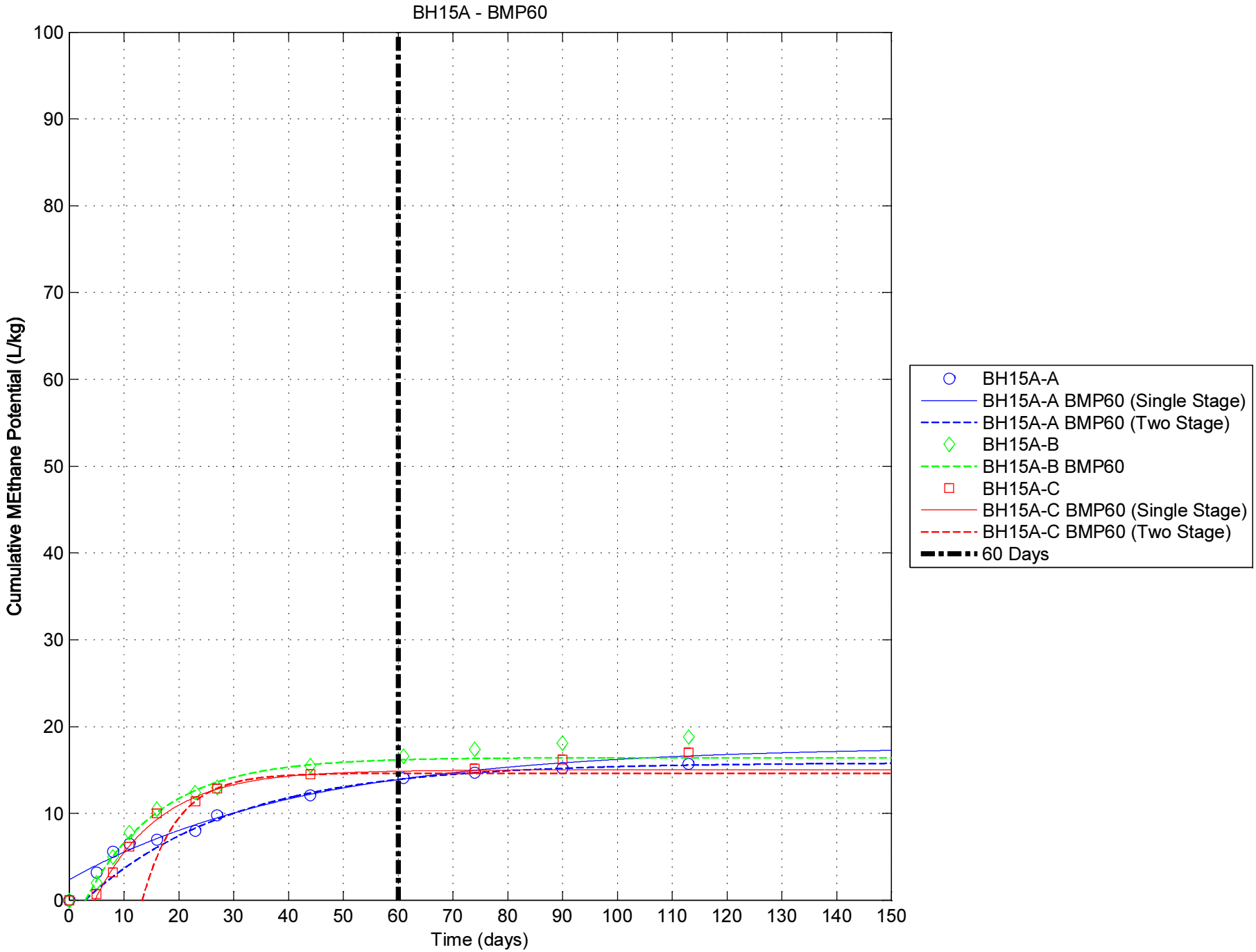

BH10A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH10A-A
BH10A-A BMP60
BH10A-B
BH10A-B BMP60
BH10A-D
BH10A-D BMP60
BH10A-E
BH10A-E BMP60
BH10A-F
BH10A-F BMP60
60 Days

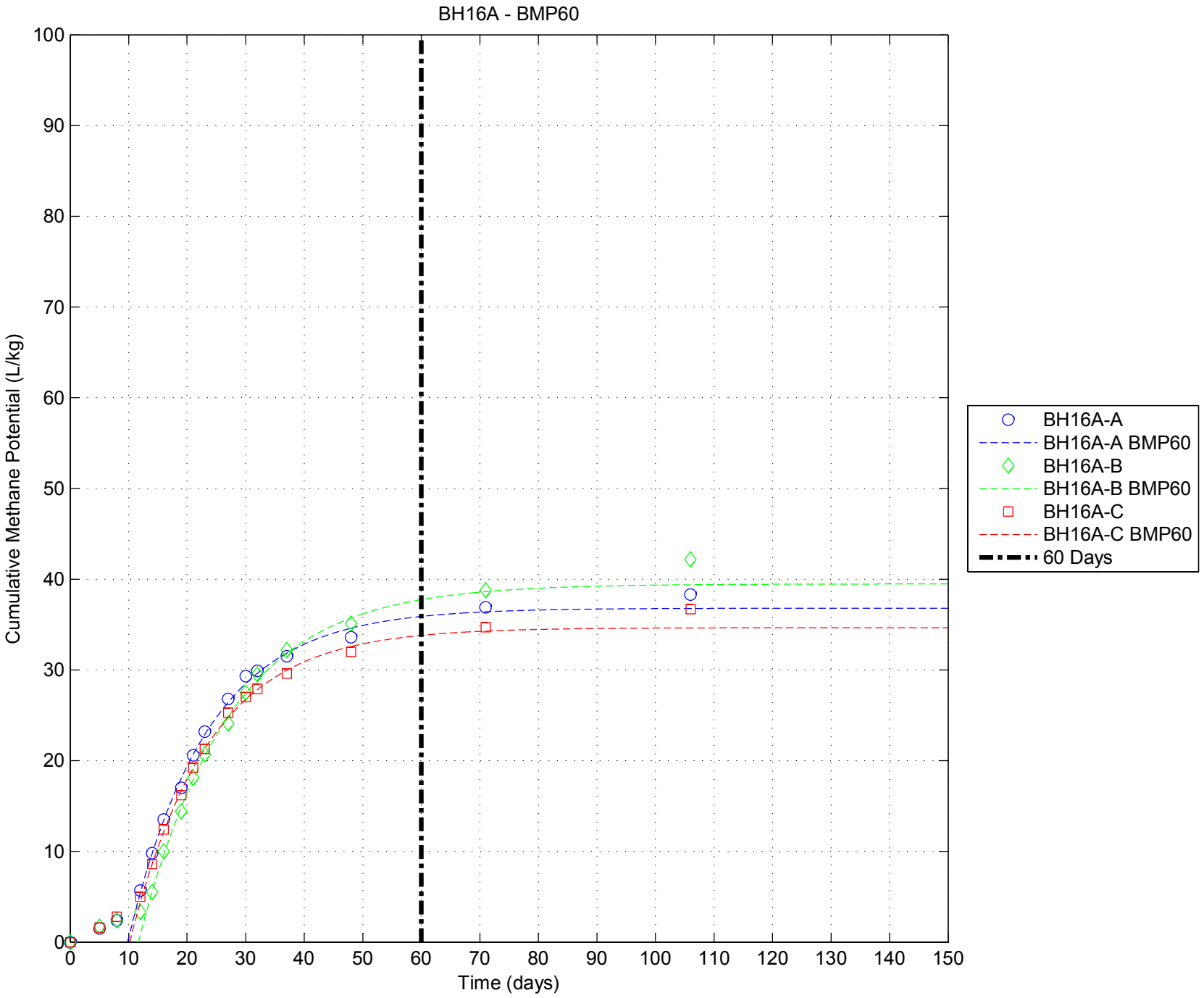

BH11A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH11A-A
BH11A-A BMP60
BH11A-B
BH11A-B BMP60
BH11A-C
BH11A-C BMP60
60 Days

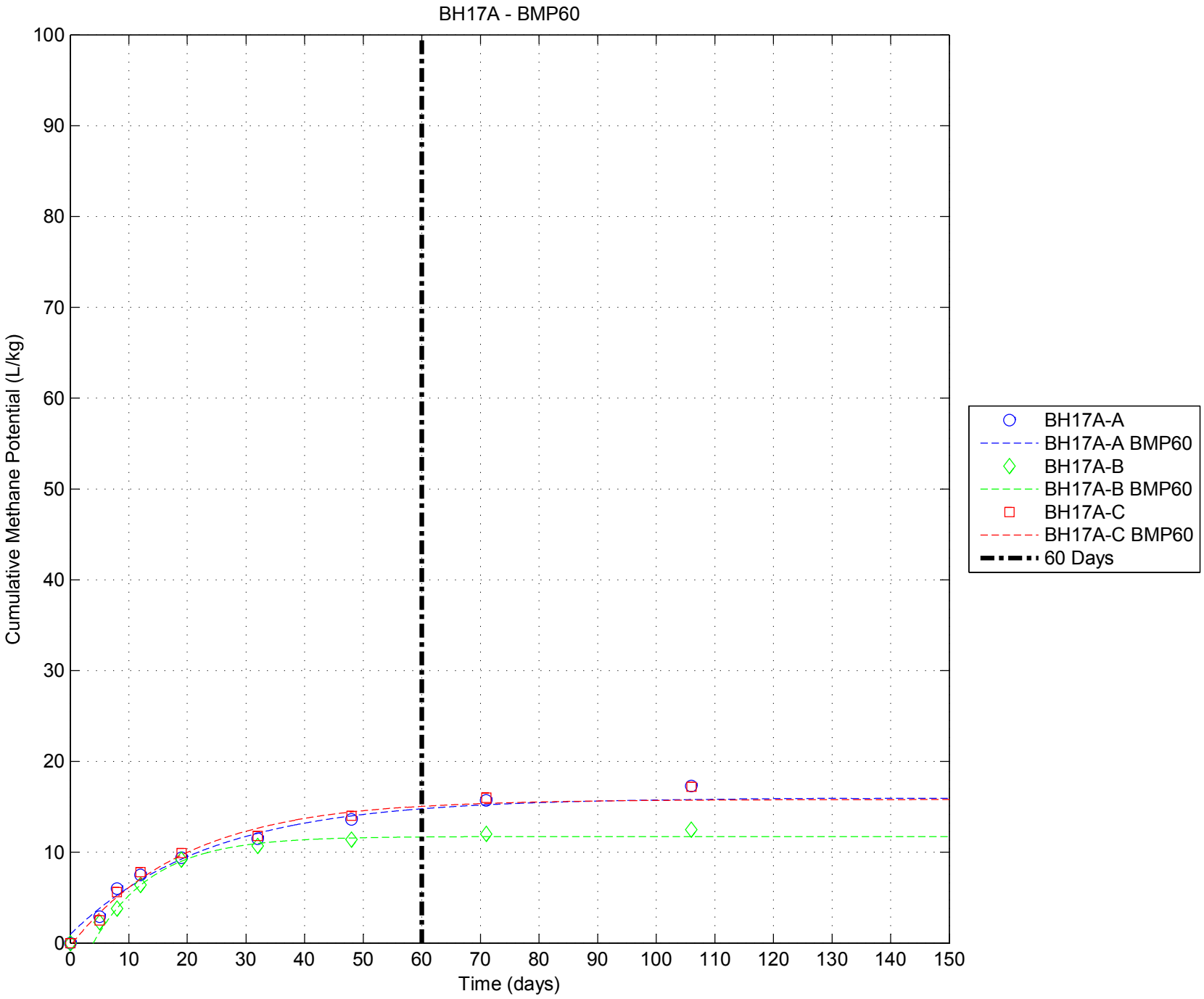

BH12A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH12A-A
BH12A-A BMP60
BH12A-B
BH12A-B BMP60
BH12A-C
BH12A-C BMP60
60 Days

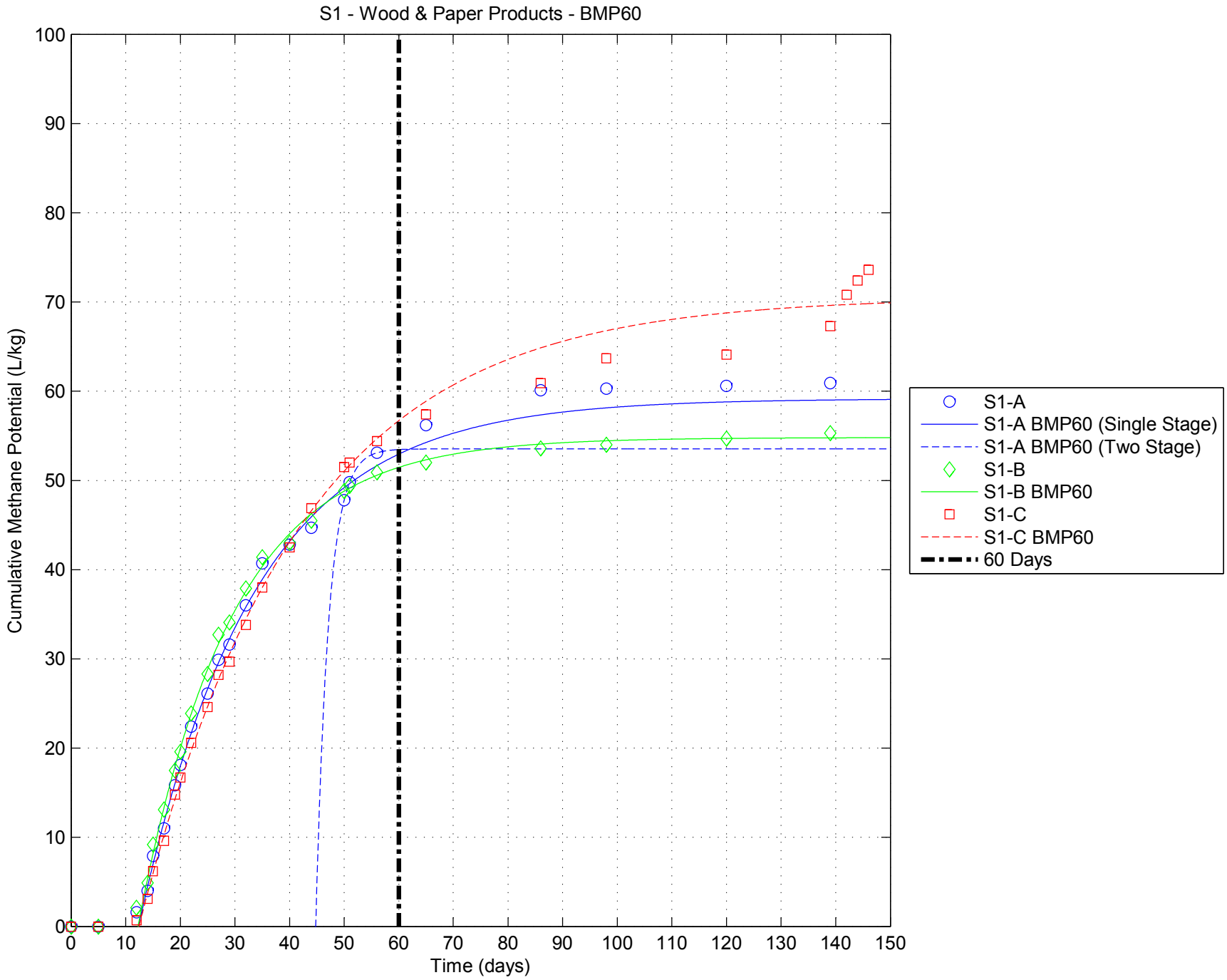

BH13A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH13A-A
BH13A-A BMP60
BH13A-C
BH13A-C BMP60
60 Days

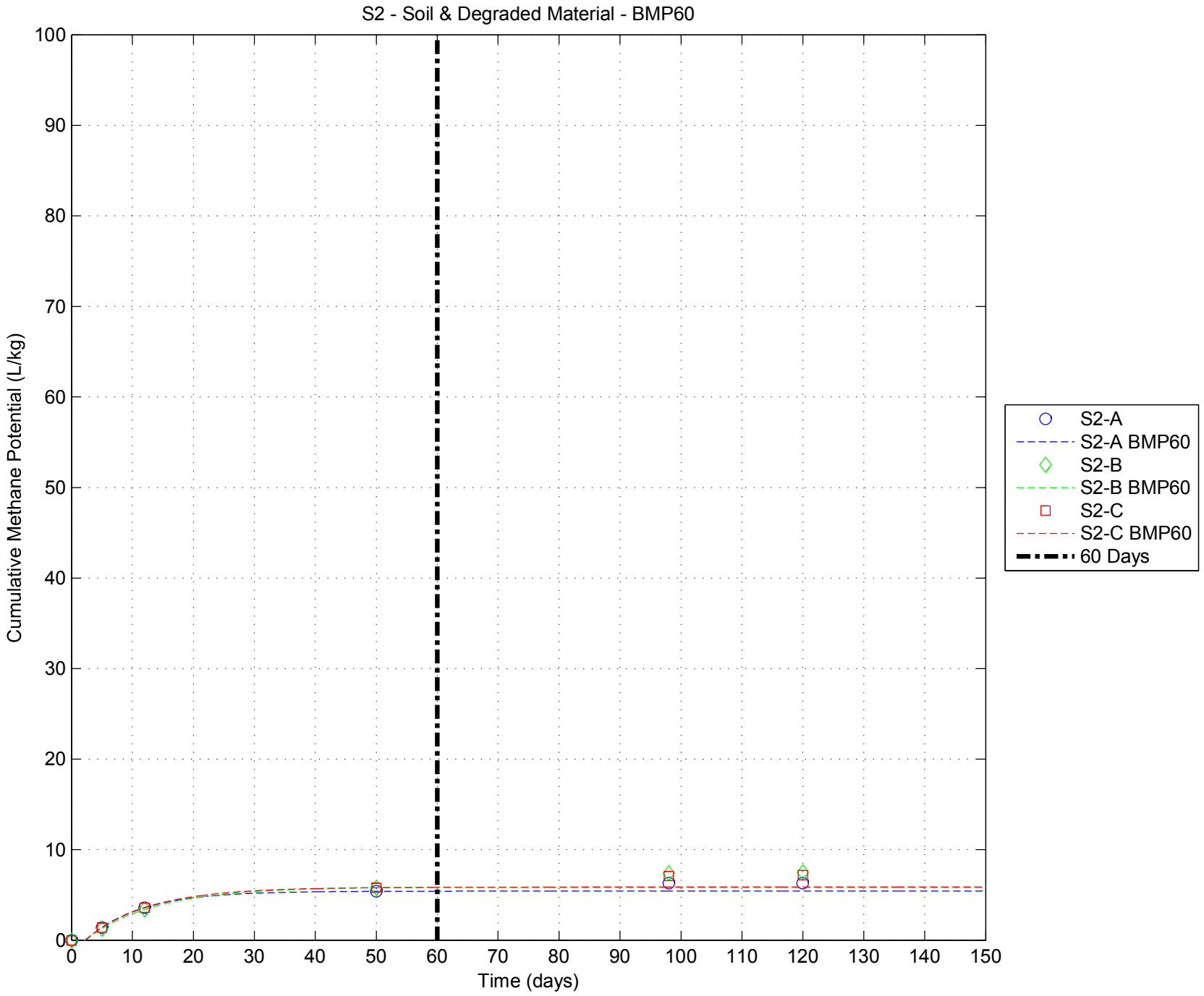

BH14A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH14A-A
BH14A-A BMP60
BH14A-B
BH14A-B BMP60
BH14A-C
BH14A-C BMP60
60 Days

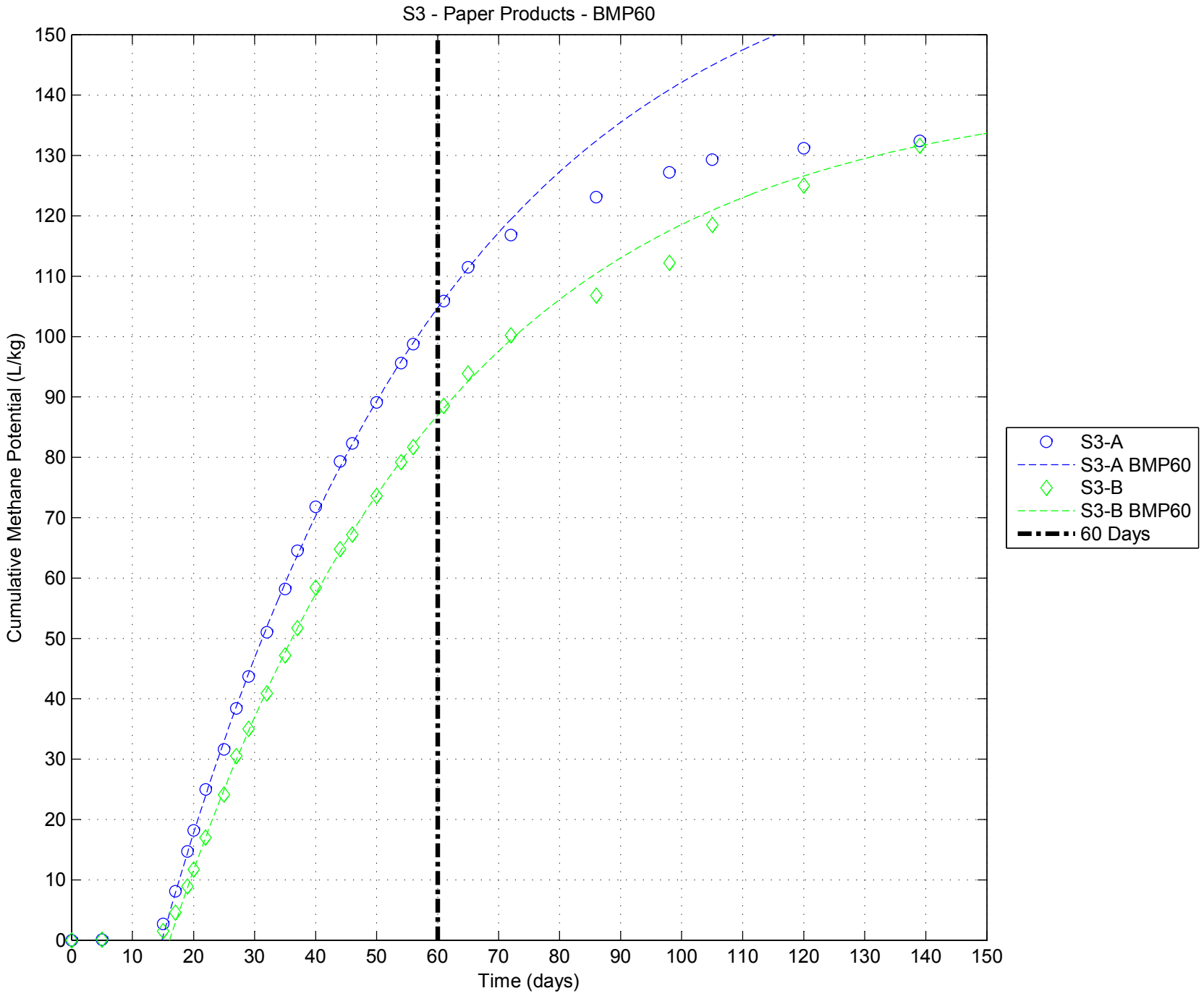

BH15A - BMP60
Cumulative MEthane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH15A-A
BH15A-A BMP60 (Single Stage)
BH15A-A BMP60 (Two Stage)
BH15A-B
BH15A-B BMP60
BH15A-C
BH15A-C BMP60 (Single Stage)
BH15A-C BMP60 (Two Stage)
60 Days

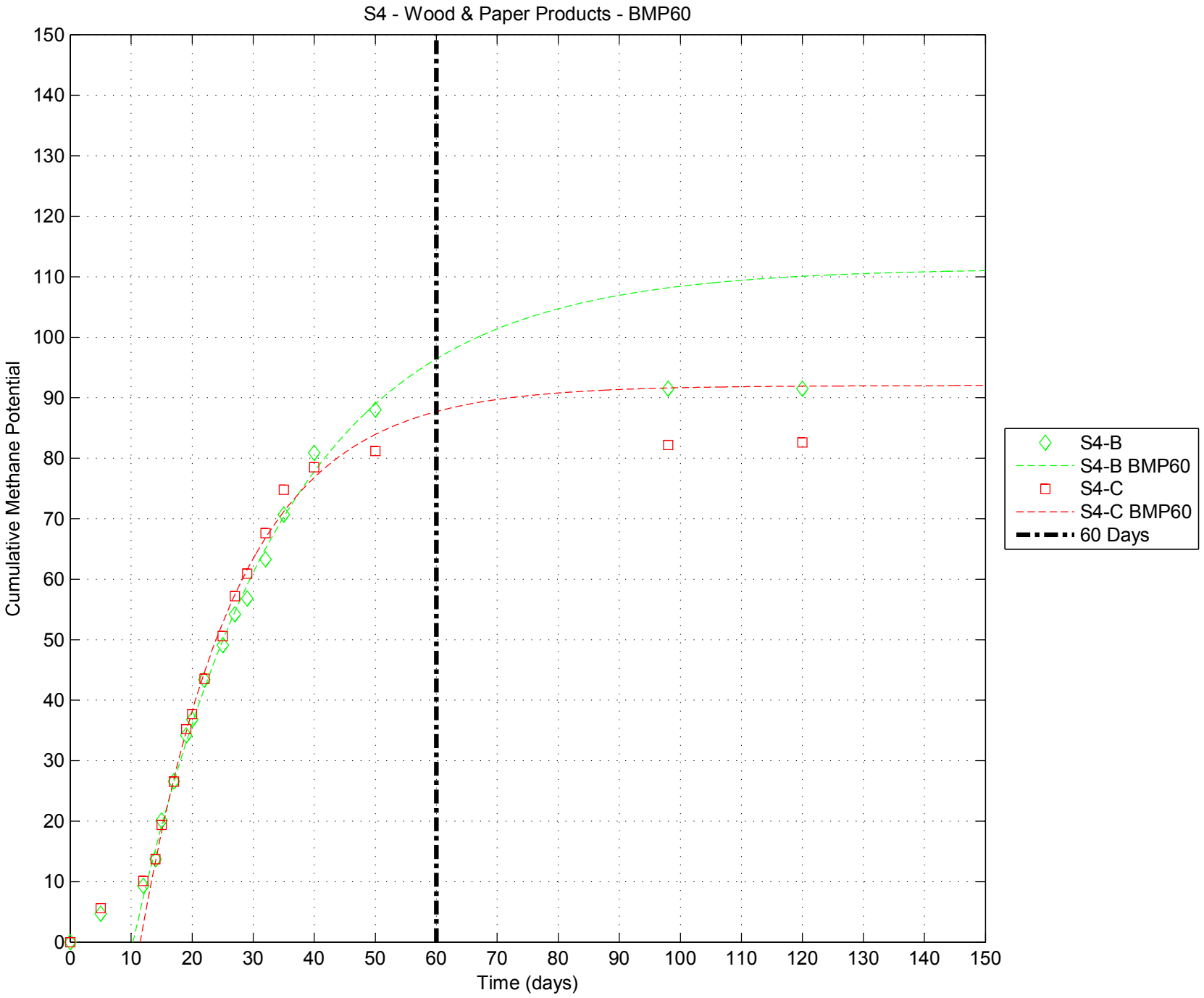

BH16A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH16A-A
BH16A-A BMP60
BH16A-B
BH16A-B BMP60
BH16A-C
BH16A-C BMP60
60 Days

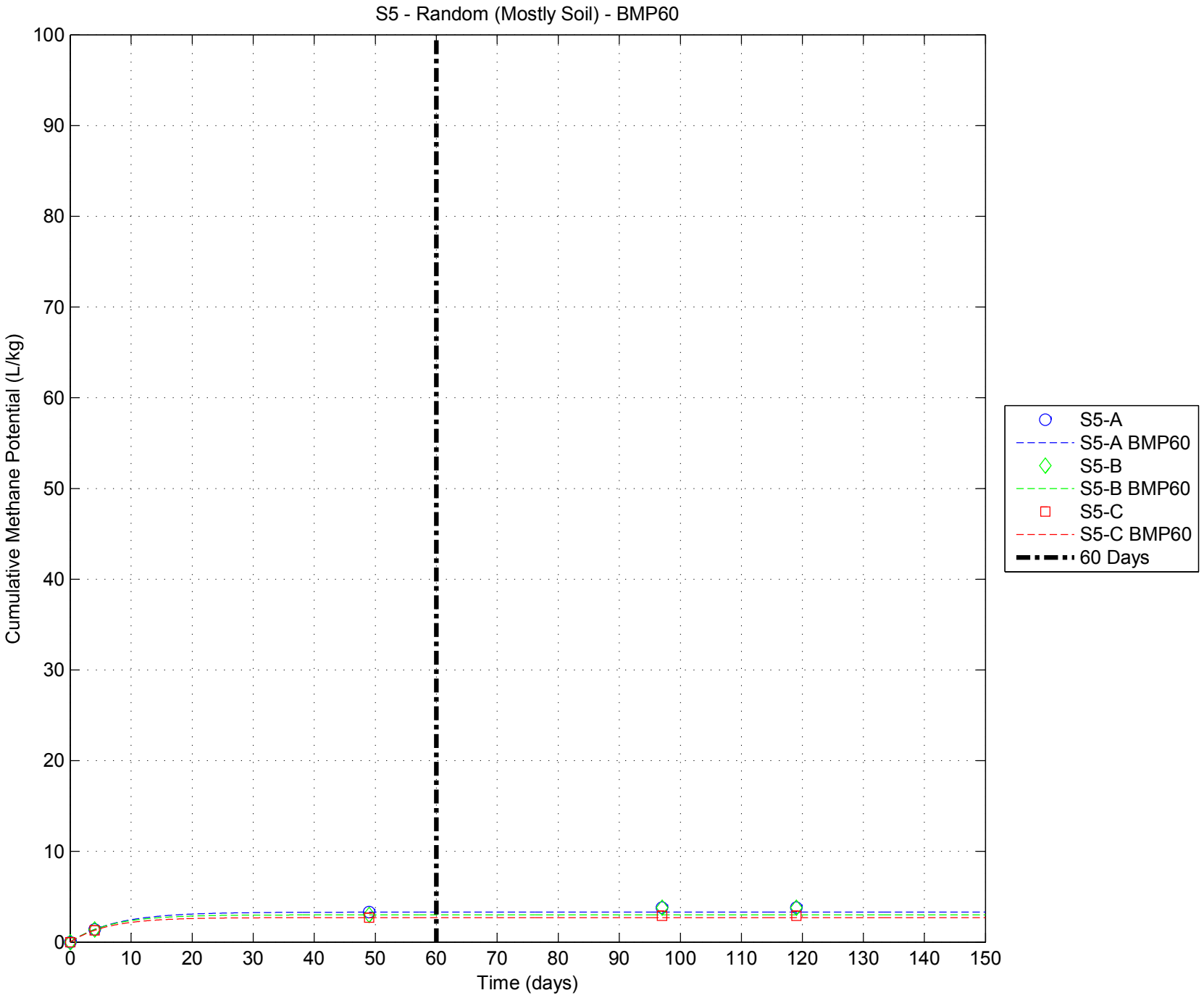

BH17A - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
BH17A-A
BH17A-A BMP60
BH17A-B
BH17A-B BMP60
BH17A-C
BH17A-C BMP60
60 Days

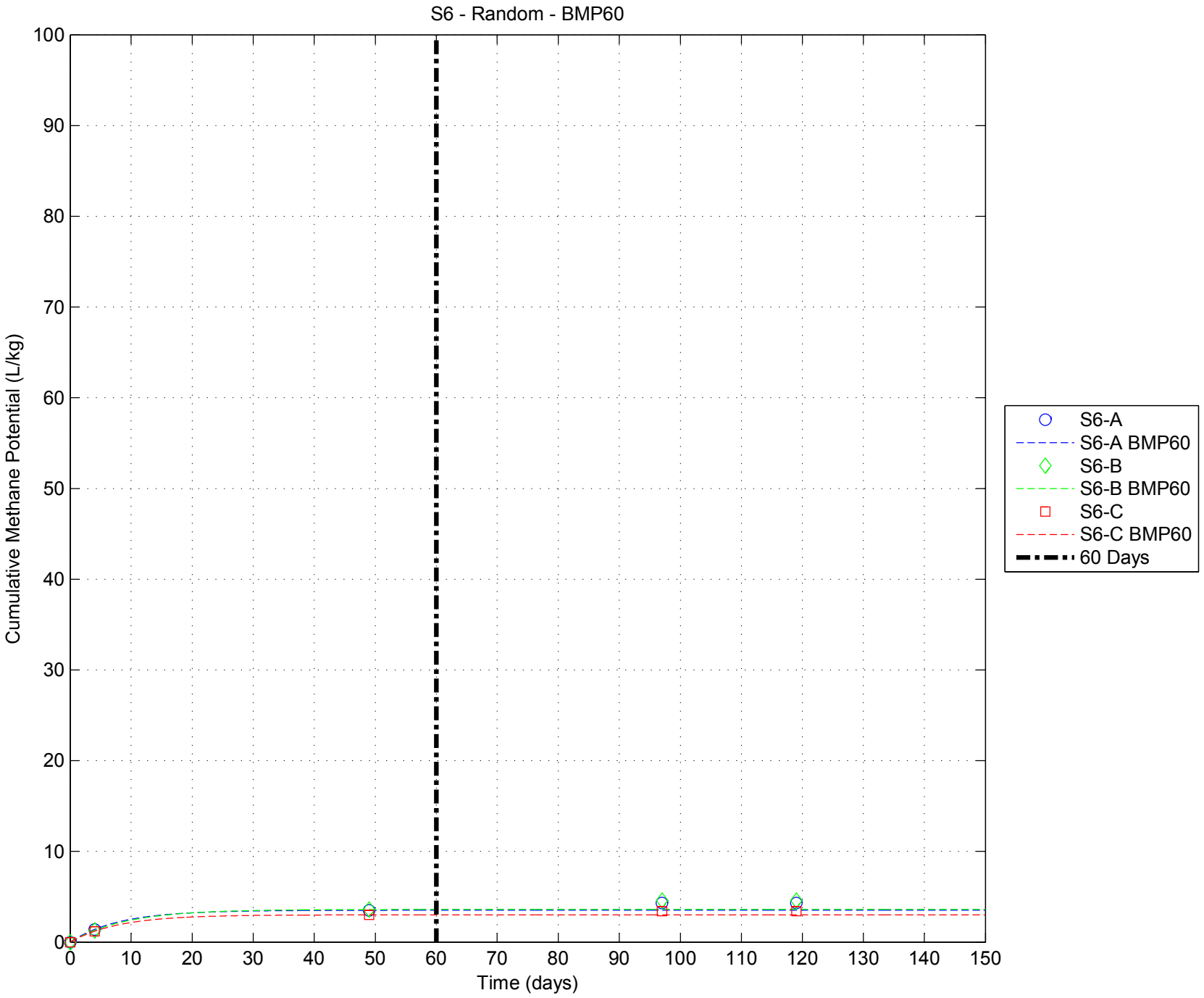

S1 - Wood & Paper Products - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S1-A
S1-A BMP60 (Single Stage)
S1-A BMP60 (Two Stage)
S1-B
S1-B BMP60
S1-C
S1-C BMP60
60 Days

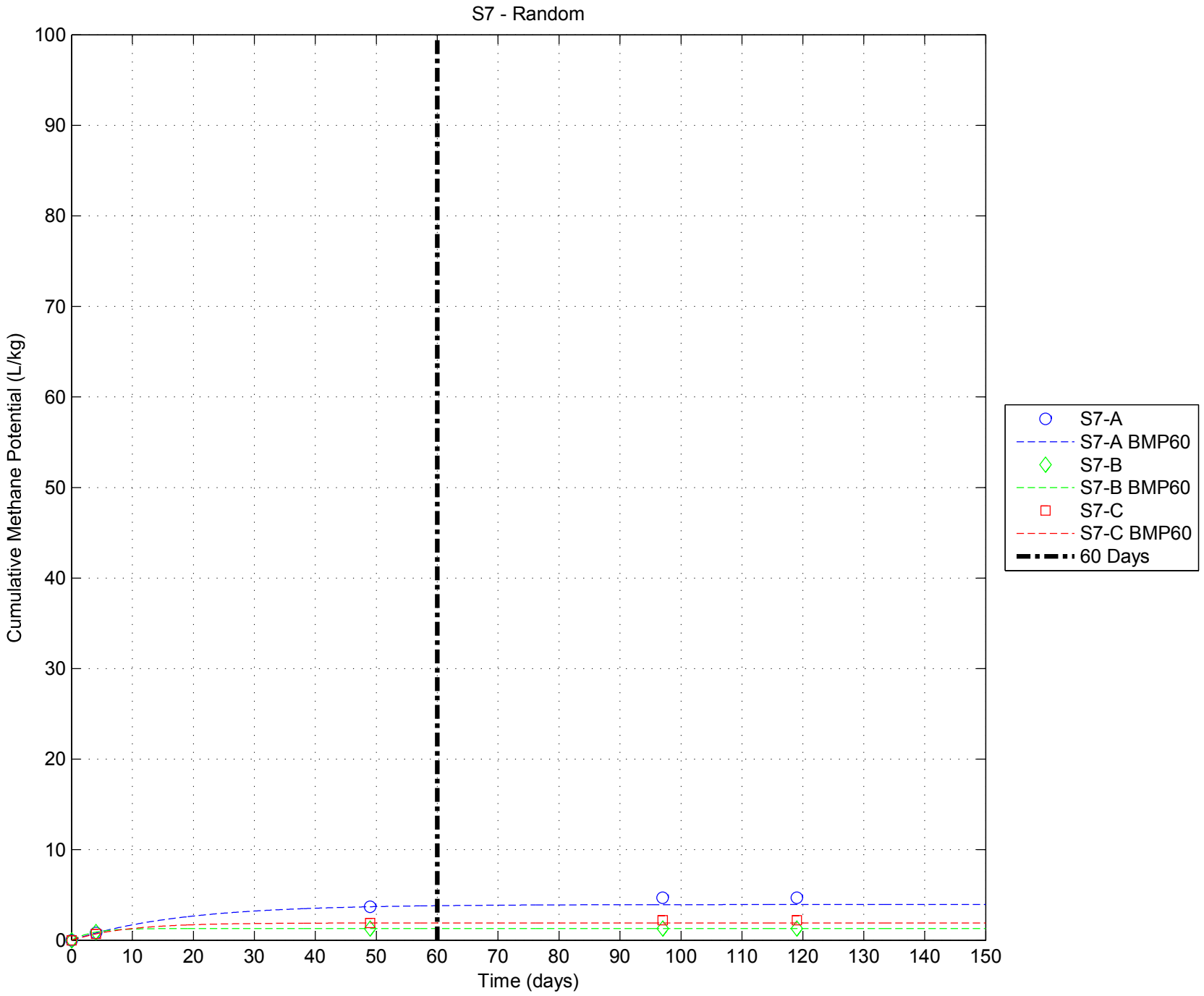

S2 - Soil & Degraded Material - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S2-A
S2-A BMP60
S2-B
S2-B BMP60
S2-C
S2-C BMP60
60 Days

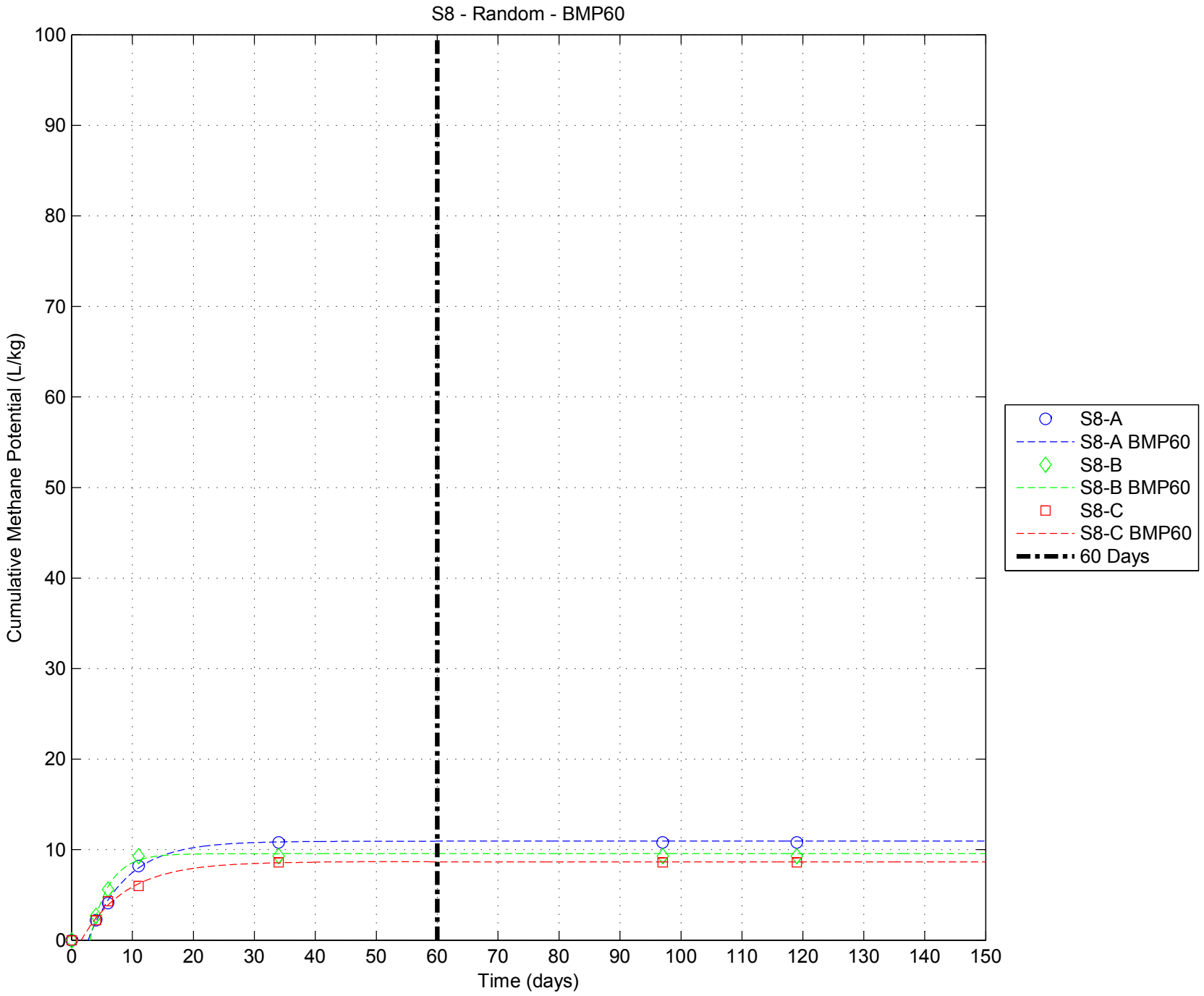

S3 - Paper Products - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S3-A
S3-A BMP60
S3-B
S3-B BMP60
60 Days

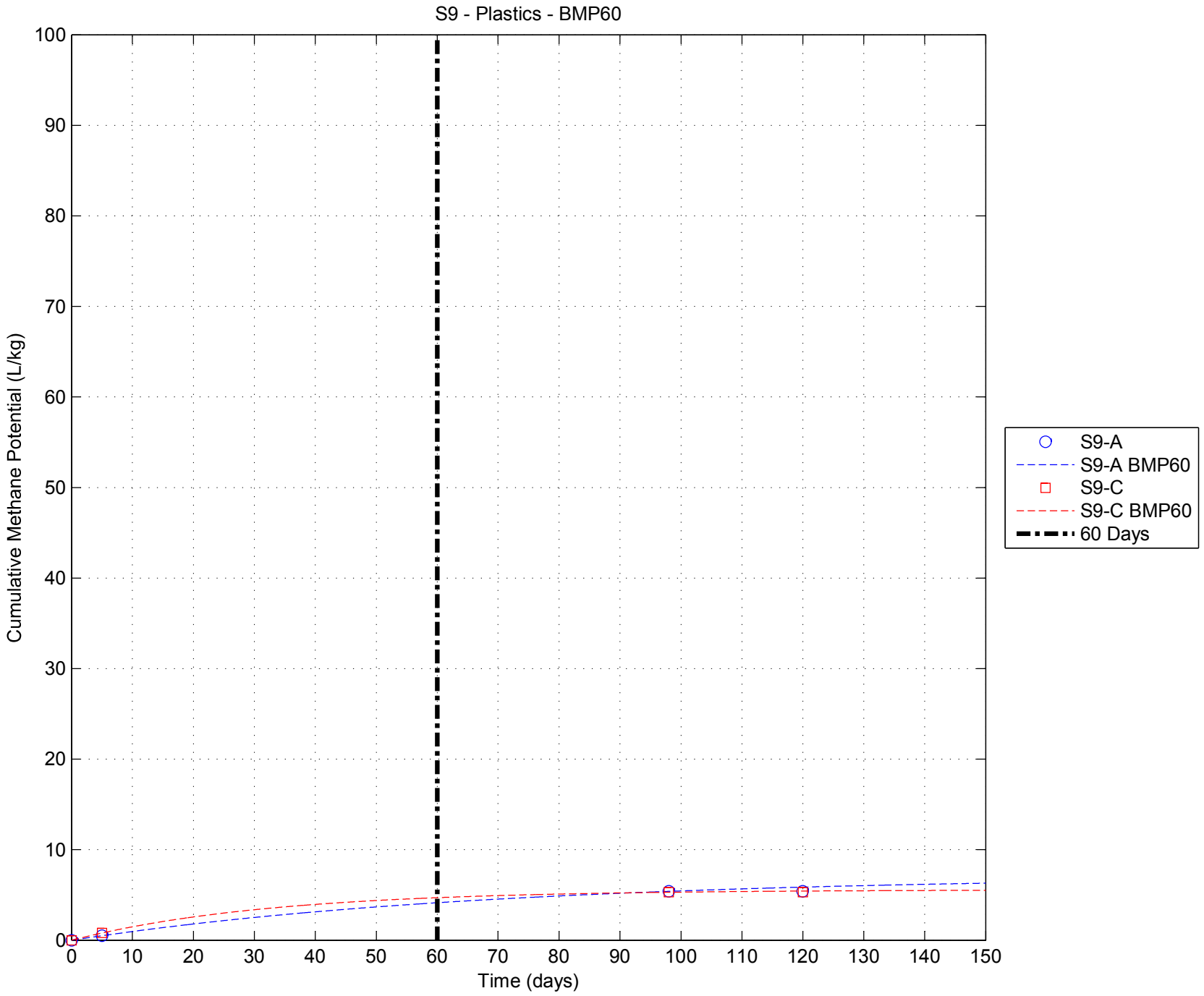

S4 - Wood & Paper Products - BMP60
Cumulative Methane Potential
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S4-B
S4-B BMP60
S4-C
S4-C BMP60
60 Days

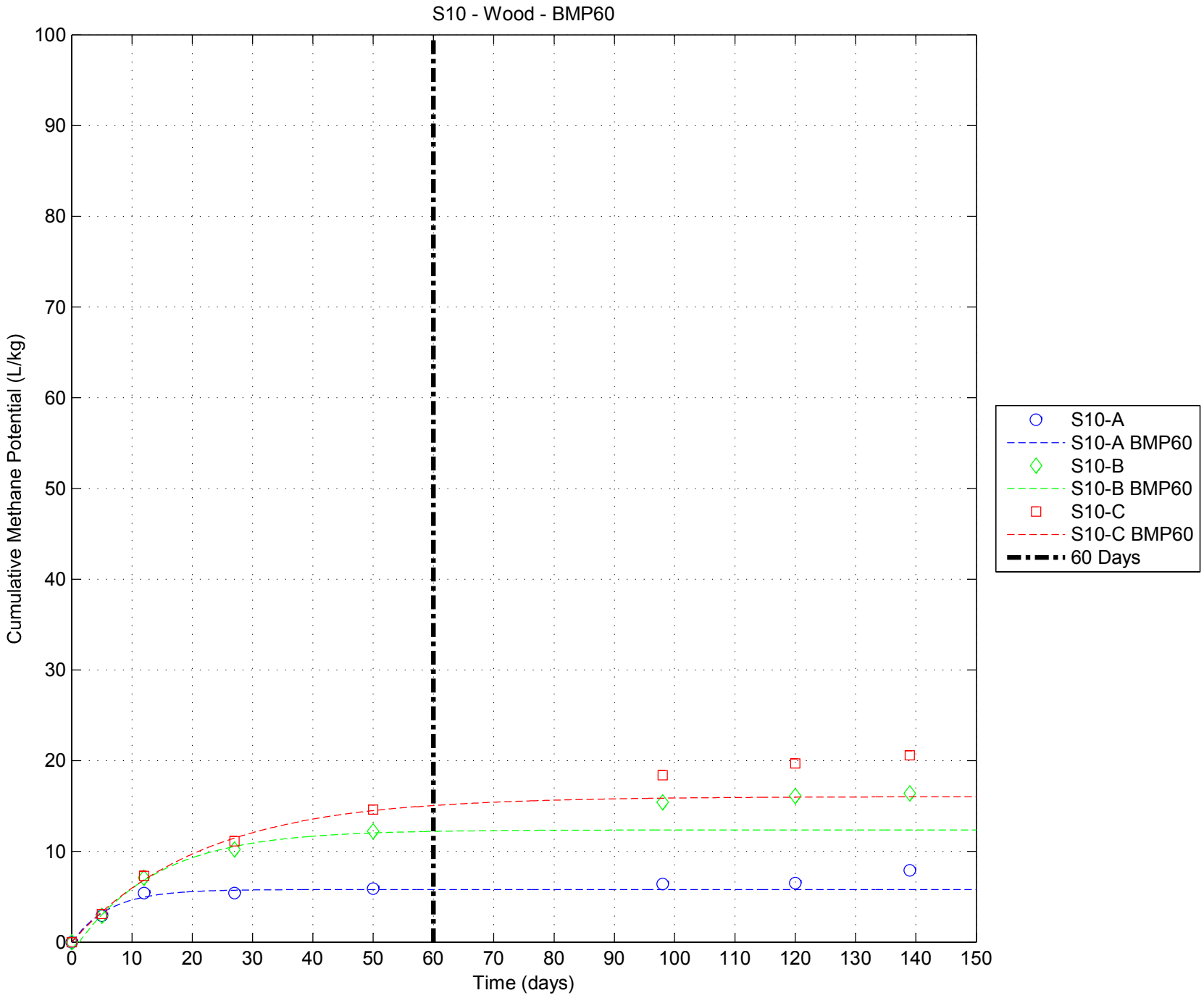

S5 - Random (Mostly Soil) - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S5-A
S5-A BMP60
S5-B
S5-B BMP60
S5-C
S5-C BMP60
60 Days

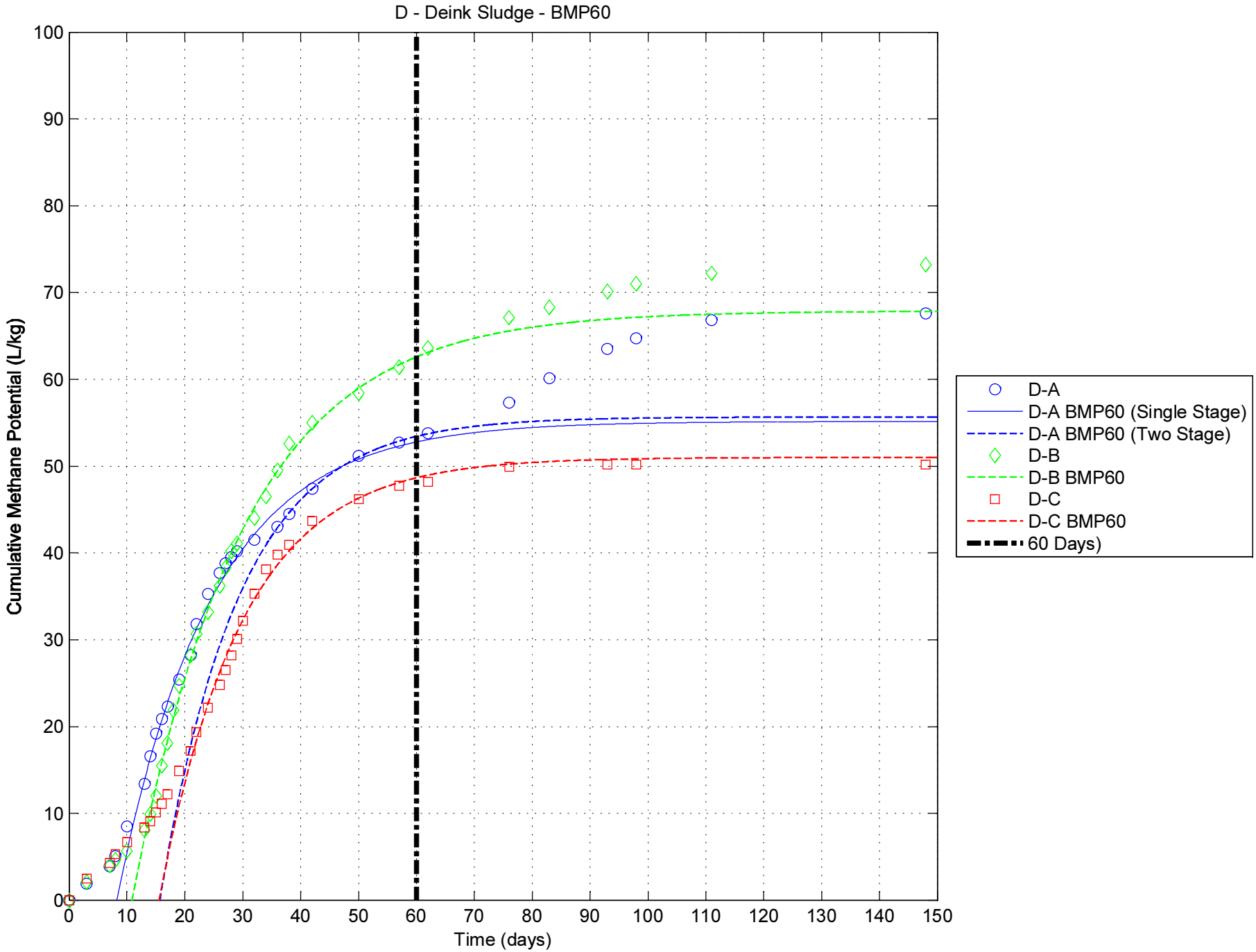

S6 - Random - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S6-A
S6-A BMP60
S6-B
S6-B BMP60
S6-C
S6-C BMP60
60 Days

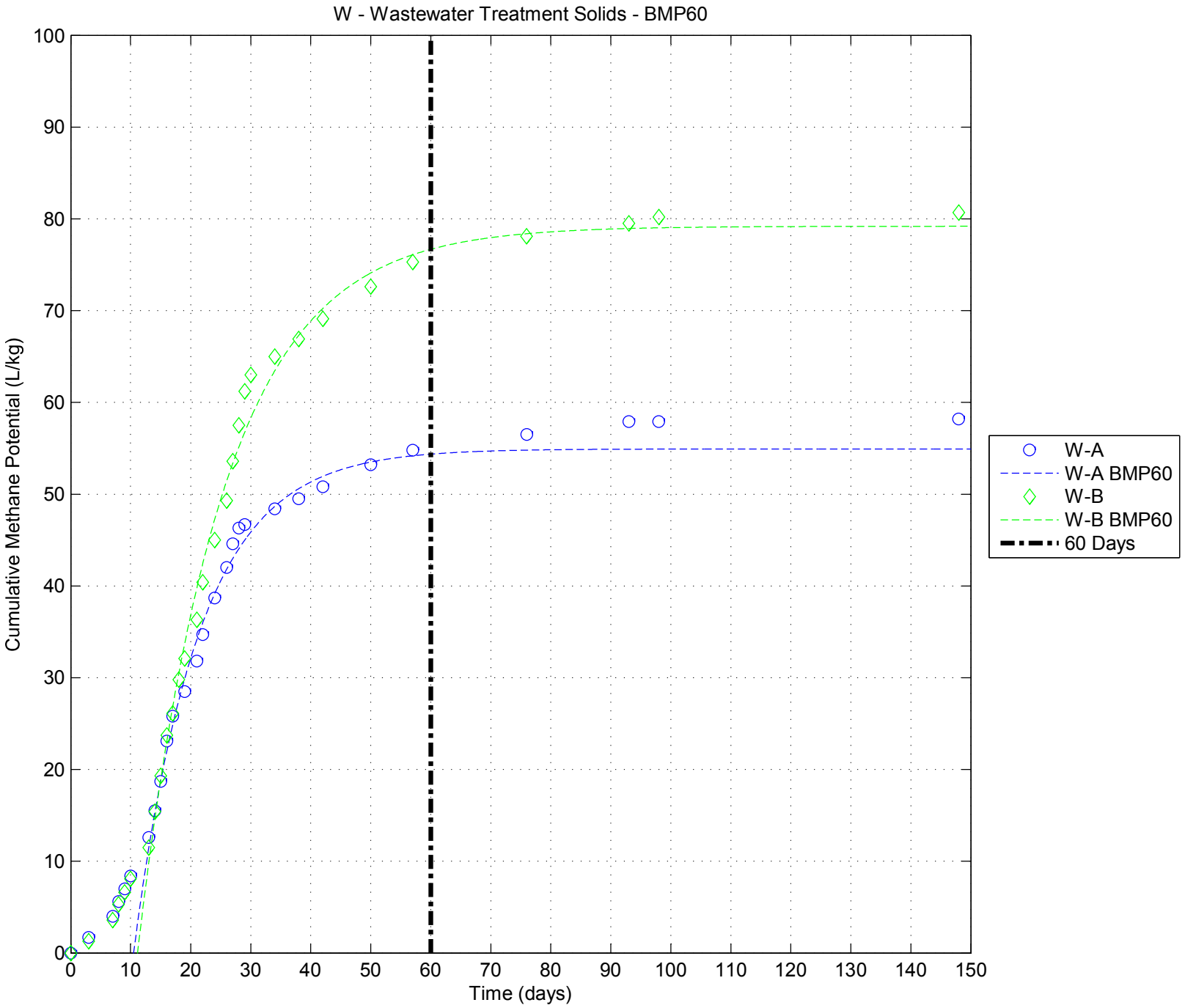

S7 - Random
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S7-A
S7-A BMP60
S7-B
S7-B BMP60
S7-C
S7-C BMP60
60 Days

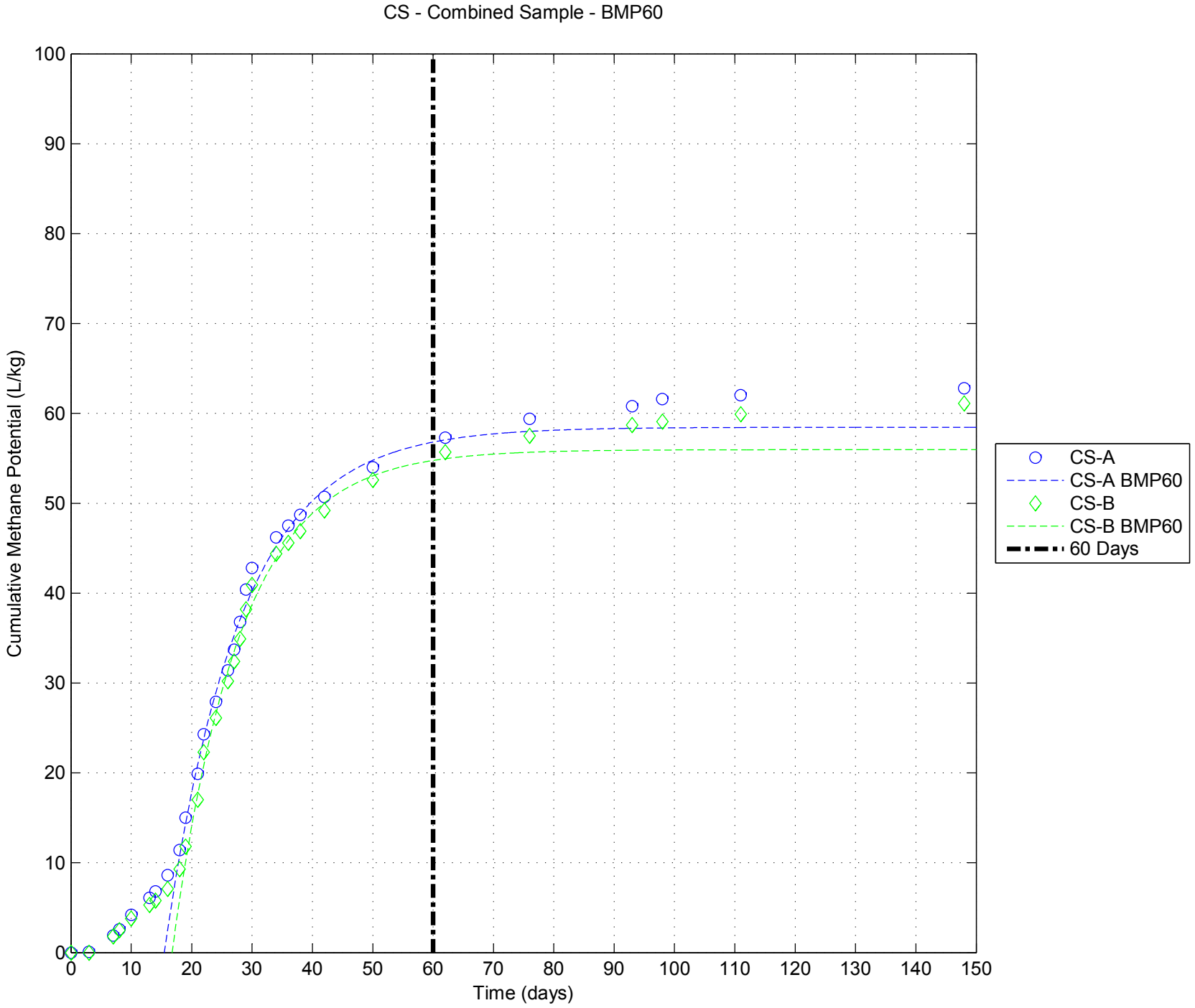

S8 - Random - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S8-A
S8-A BMP60
S8-B
S8-B BMP60
S8-C
S8-C BMP60
60 Days

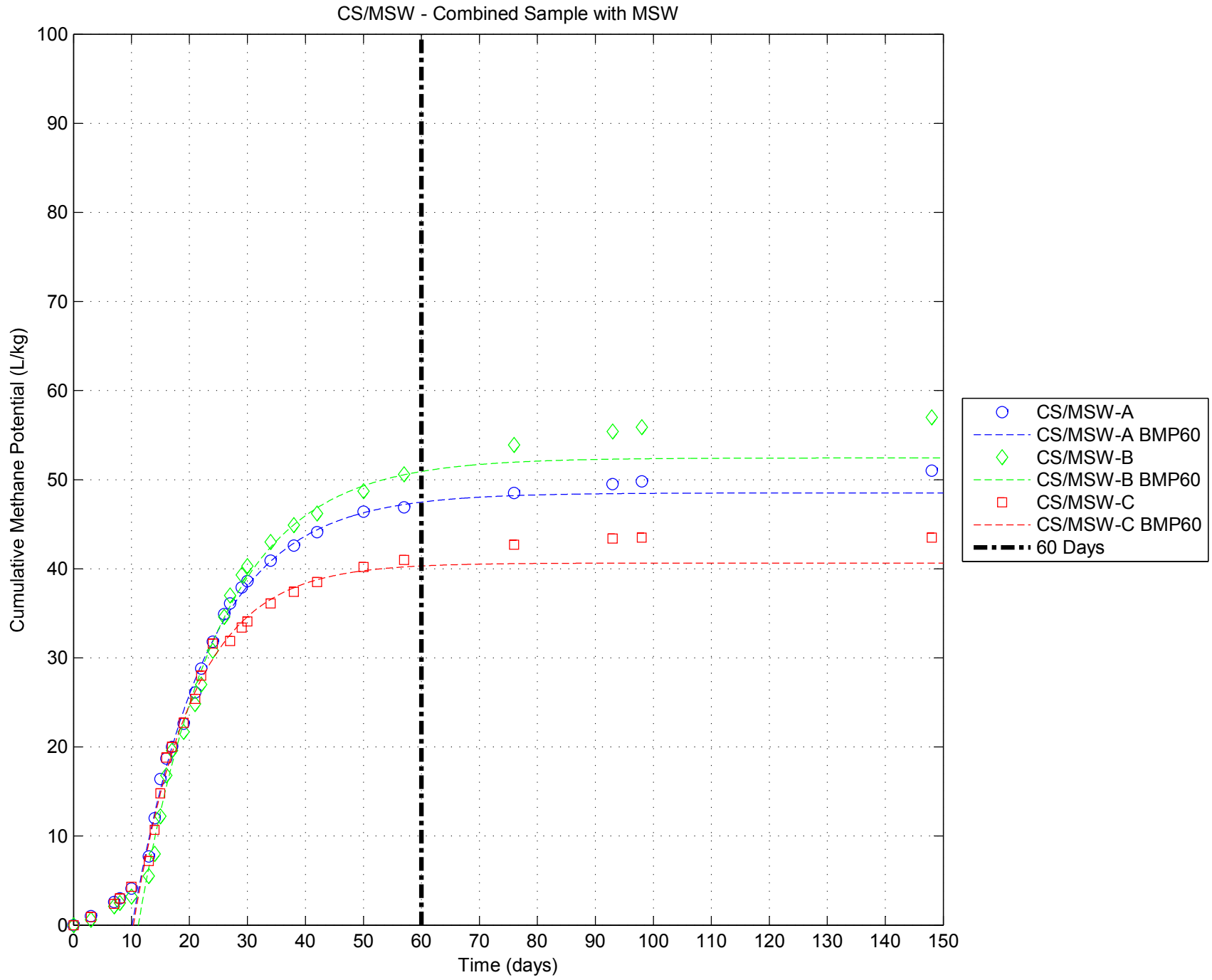

S9 - Plastics - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S9-A
S9-A BMP60
S9-C
S9-C BMP60
60 Days


S10 - Wood - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
S10-A
S10-A BMP60
S10-B
S10-B BMP60
S10-C
S10-C BMP60
60 Days


D - Deink Sludge - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
D-A
D-A BMP60 (Single Stage)
D-A BMP60 (Two Stage)
D-B
D-B BMP60
D-C
D-C BMP60
60 Days)


W - Wastewater Treatment Solids - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
W-A
W-A BMP60
W-B
W-B BMP60
60 Days


CS - Combined Sample - BMP60
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
CS-A
CS-A BMP60
CS-B
CS-B BMP60
60 Days


CS/MSW - Combined Sample with MSW
Cumulative Methane Potential (L/kg)
Time (days)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
CS/MSW-A
CS/MSW-A BMP60
CS/MSW-B
CS/MSW-B BMP60
CS/MSW-C
CS/MSW-C BMP60
60 Days